N71-33004

NASA-CR-72869
WANL-PR-222-001
December, 1970

DEVELOPMENT OF LARGE DIAMETER T-111 (Ta-8W-2Hf) TUBING

FINAL REPORT

by

D. R. Stoner and R. W. Buckman Jr.

Westinghouse Astronuclear Laboratory

Prepared for

National Aeronautics and Space Administration

NASA- Lewis Research Center

Contract NAS 3-10602

J. A. Milko - Project Manager

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Astronuclear Laboratory

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CONTRACT NAS 3-10602

NASA Lewis Research Center
Cleveland, Ohio
J. A. Milko, Project Manager
Materials and Structures Division

FOREWORD

The work described in this report was carried out by personnel of the Westinghouse Astronuclear Laboratory under Contract NAS 3-10602, "Development of Large Diameter T-111 Tubing," for the NASA-Lewis Research Center. The work was administered at the Astronuclear Laboratory by Messrs. R. T. Begley and R. W. Buckman, Jr. Mr. J. A. Milko was the NASA Project Manager for this program. Mr. D. R. Stoner was responsible for the direction of the experimental aspects of this program at the Westinghouse Astronuclear Laboratory.

The authors gratefully acknowledge the assistance of the many people who contributed to the success of this program: at Lewis Research Center, T. A. Moss and R. L. Davies for their guidance, and at Westinghouse, G. G. Lessmann for advice on welding and post weld heat treating; R. Sprecace for gas tungsten arc welding; K. Galbraith for metallography, and E. Vandergrift for mechanical testing. Also the success of this largely subcontracted program is due in large measure to the cooperation of the following companies:

Melting and Vacuum Annealing	-	Wah Chang, Albany, Oregon
Forging	-	Industrial Forge, Albany, California
Plate Rolling	-	Stellite Div. Cabot Corporation*, Kokomo, Indiana
Extrusion	-	Canton Drop Forge, Canton, Ohio
Tube Reducing	-	Timken, Wooster, Ohio
Tube Shell Forming	-	Swepco Corporation, Clifton, New Jersey
Tube Welding	-	Mech-Tronics, Chicago, Illinois - Div. of Fansteel Metallurgical Corporation

* Formerly Haynes Stellite Div., Union Carbide

ABSTRACT

Large diameter T-111 (Ta-8W-2Hf) tubing was processed from 5 1/2 inch x 1/2 inch wall seamless and welded tube shells using current state-of-the-art commercial metal working equipment. Twenty feet each of 4 1/4 inch OD x 1/8 inch wall and 3 inch OD x 0.080 inch wall, weighing a total of 400 pounds with a maximum single continuous length of eleven feet was produced. Tube roundness and dimensional control were better than commercial practice. Both the seamless and weld-reduced tubing were made from a single 2400 pound T-111 ingot. There were no significant differences between the room temperature tensile properties or the 2400°F creep rupture behavior of the seamless and the weld-reduced tubing, and the values obtained are comparable to those reported for T-111 sheet product.

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I. INTRODUCTION AND SUMMARY

This report describes the results of a program where the principal objective was to fabricate large diameter T-111* tubing from seamless and welded tube shells using current state-of-the-art commercial metal working equipment and practices. The differences in processing tubing from seamless and welded tube shells were evaluated as well as the room and elevated temperature mechanical properties of the final product.

Using available commercial metal working equipment, and by exercising strict controls over each stage of processing, high quality T-111 tubing in two different sizes was successfully produced from both the seamless and welded T-111 tube shells. Twenty feet each of 4 1/4 inch OD x 1/8 inch wall and 3 inch OD x 0.08 inch wall, weighing approximately a total of 400 pounds with a maximum single continuous length of eleven feet, were produced.

Tube roundness and dimensional control were better than commercial practice with roundness within 0.002 inch, diameter 0.005 inch, and wall thickness within 0.003 (4%) and 0.010 inch (8%) respectively of the target size for the 3 and 4 1/4 inch diameter tubing.

After a final recrystallization anneal of one hour at 3000°F, typical properties of specimens taken parallel to the tube axis from both tube sizes were as follows:

Room Temperature	- 0.2% Yield Strength - 75,000 psi Ultimate Tensile Strength - 90,000 psi Elongation in one inch - 40%
2400°F	- Stress for Rupture in 100 hours - 18,000 psi

*T-111 is a tantalum base alloy having the nominal composition in weight percent of Ta-8W-2Hf.

There were no significant differences observed between the properties of the seamless and the weld*redrawn tubing, and the values obtained are comparable to those reported for T-111 sheet product.

Tubing was produced from a single 2400 lb homogeneous T-111 ingot consolidated by a combination of electron beam and consumable electrode vacuum arc melting. This ingot, which provided all the program material, was the largest single tantalum alloy ingot ever produced. The as-cast ingot was lathe conditioned and then sectioned to provide two billets weighing 470 and 920 pounds respectively. Primary working of the as-cast billets was by a combination of hot extrusion and/or forging. The fabrication sequence for producing the seamless and welded tube shells is outlined in Figure 1-1. The cast T-111 extrusion billets were contained within an evacuated mild steel cladding which provided protection during heating and the subsequent extrusion. The solid extrusion was hammer forged to bar which was subsequently rolled to plate. After annealing, the 0.425 inch thick plate was formed into a cylinder which was then single pass electron beam welded. The 5 1/2 inch diameter seamless and welded tube shells were then tube reduced at room temperature to the desired final sizes of 4 1/4 inch and 3 inch diameter with wall thicknesses of 1/8 inch and 0.080 inches respectively. Strict controls were exercised over each of the critical processing areas such as the high temperature working, conditioning, cleaning, and pickling operations to ensure generation of a sound product. The process and final annealing treatments were done in a vacuum furnace at $3000^{\circ}F$ at a pressure below 5×10^{-5} torr. Tantalum foil wrapping of the workpiece was also utilized as an additional precaution against extraneous contamination during vacuum annealing.

The feasibility of producing large diameter T-111 tubing, successfully demonstrated during the course of this investigation, was due in large measure to the cooperation of the American metal working industry and their enthusiasm and technical input is gratefully acknowledged.

*Single pass electron beam welding was used to produce defect free tube shell weldments. (As a result of work on this program, it was discovered that T-111 undergoes underbead fissuring during multipass welding of thick sections, and this behavior is discussed in Appendix 3.)

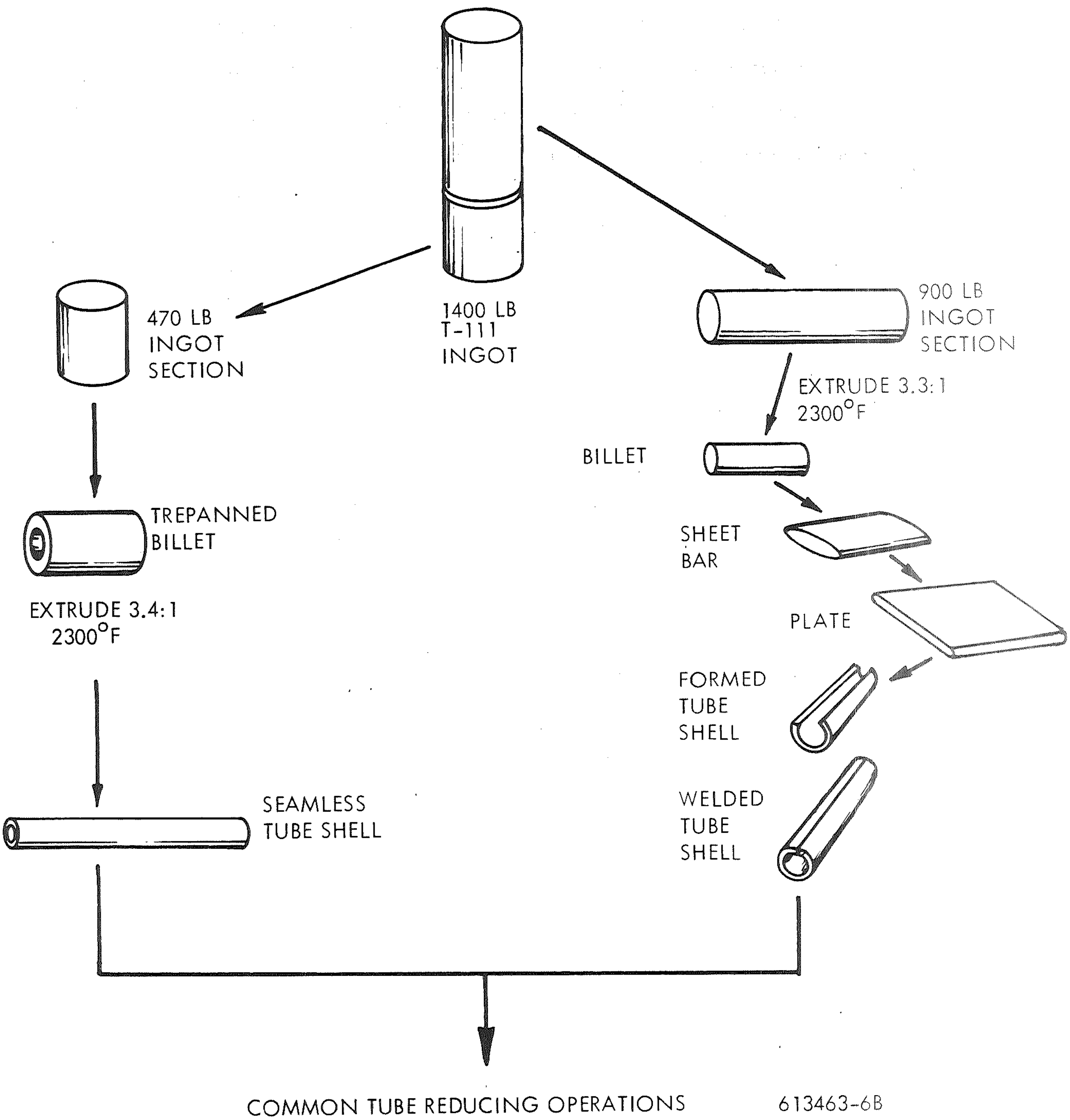


Figure 1-1. Tube Shell Fabrication Sequence

A listing of the participating personnel and a description of the equipment used at each of these companies is in Appendix 1. Some sense of the coordination that was required in scheduling and material handling is illustrated by the 46,000 air miles traveled by the T-111 material from ingot stage through the final tubing size.

2. BACKGROUND

The NASA Lewis Research Center has designed systems to convert thermal energy to electric power using Brayton and Rankine thermodynamic cycles or by direct conversion. The major design objective of high thermal efficiency at minimum system weight is realized by operating at the highest possible temperature and at moderate working fluid pressures. In this respect, alkali metals are excellent heat transfer and working fluids, while refractory metal alloys, combining superior high temperature strength and excellent corrosion resistance in alkali metals, are uniquely suited for system structures. The most severe shortcomings of refractory metals, poor oxidation resistance and adverse ductility response to atmospheric contamination, are avoided in the high vacuum space environment.

Of the class of fabricable and weldable tantalum base alloys under development since the early 1960's, T-111 (Ta-8W-2Hf) has demonstrated an optimum combination of high temperature creep strength, fabricability, weldability, thermal stability, and resistance to alkali metal corrosion. This combination of properties has resulted in selection of T-111 as the reference material for advanced space power systems. Relatively large quantities of T-111 have been processed as plate, sheet, and small diameter tubing for construction of and evaluation in liquid alkali metal loops and as encapsulation materials for radioisotopes[1,2*].

An important step in the utilization of any promising alloy though is to fabricate and evaluate the properties of full size components. Towards this end, work was initiated to develop and document the fabrication techniques for producing 4 1/4 and 3 inch diameter T-111 tubing using available metal working equipment. This size tubing is required as piping in liquid metal cooled reactors and inlet and outlet tubing for alkali metal heat exchangers. The tubing produced in this development program would permit the construction of full scale corrosion test and pressure test models and would guide welding and fabrication techniques for the eventual hardware systems.

*References

A prime requirement throughout the program was the need to use existing metal working and fabrication techniques with special precautions in areas critical to refractory metal alloys such as melting, heat treating, welding, and chemical cleaning. The expediency requirement was justified in view of the cost and time required to develop special equipment, dies, and techniques for 5 1/2 inch diameter, heavy wall tantalum alloy tubing.

The capacity of available melting and primary metal working equipment limits the total deformation from the as-cast ingot to the required large starting tube shell size (5 1/2 inch OD x 1/2 inch wall) thereby restricting the degree of refinement of the metallurgical structure. Thus fabrication of tube shells by welding cylinders formed from rolled plate was investigated as an alternate fabricating method. This technique is readily adaptable to weldable alloys with demonstrated ductile weld properties as T-111. Also this technique has the added advantage for producing limited quantities of tubing in that sheet and plate processing is usually more well defined for experimental alloys than seamless tube shell extrusion parameters. To eliminate heat to heat variability, material for both the welded and seamless tube shells was to be obtained from the same large ingot. A minimum of 1400 lbs. of conditioned ingot was required for the program which included a 50% contingency allowance in the welded tube shell plate.

3. SEAMLESS TUBE SHELL PROCESSING

A single tube shell was produced by extruding a hollow ingot at 2300°F over a fixed mandrel. The extrusion was machined to final tube shell configuration and vacuum annealed to provide a fine grain recrystallized structure.

3.1 Ingot Consolidation

The approximately 600 lbs. of T-111 required for the seamless tube shell was obtained from a 2340 lb., 52 inch long, 10 inch diameter ingot. This ingot, the largest tantalum alloy ingot melted to date, furnished starting material for the entire program, about 1500 lbs. in total. The ingot was prepared and melted by the Wah Chang Corporation* according to the sequence shown in Figure 3-1. This sequence consisted of double electron beam melting pressed alloy powders to produce a 4 inch diameter consumable tantalum-tungsten alloy electrode. Hafnium additions and adjustments in tungsten levels were made to this electrode which was consumable electrode double vacuum arc melted to produce a 10 inch diameter ingot. The double electron beam melting served to remove volatile impurities and the double consumable electrode melt, with complete alloy additions, assured homogeneity in the completed ingot. The 2340 lb., 10 inch diameter ingot required 18,000 amps and 38 volts of direct current power to complete the melt in 2 hours. A stirring coil was used to continuously agitate the molten pool and a vacuum of 1 micron was maintained during the melting operation.

After cropping the top and bottom, the machine conditioned ingot was ultrasonically inspected using a contact technique. No evidence of internal pipe or porosity was detected which was confirmed by subsequent processing. The ingot was sectioned as shown in Figure 3-2 to provide a 15 inch length for the seamless tube shell and a 27 inch length for producing the plate for welded tube shell plus a bottom section for a nonrelated contract. The chemical analyses

*See Appendix 1

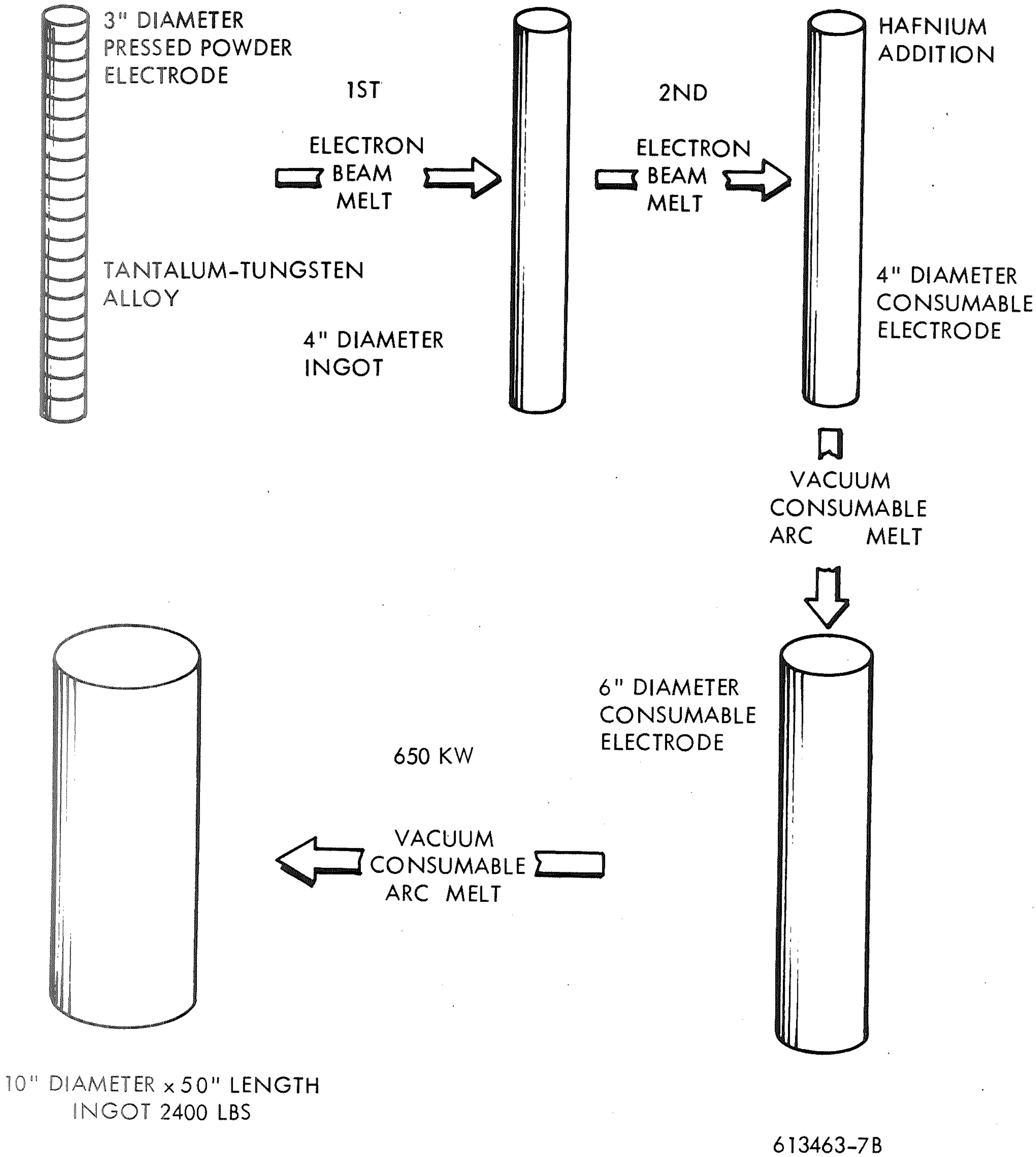
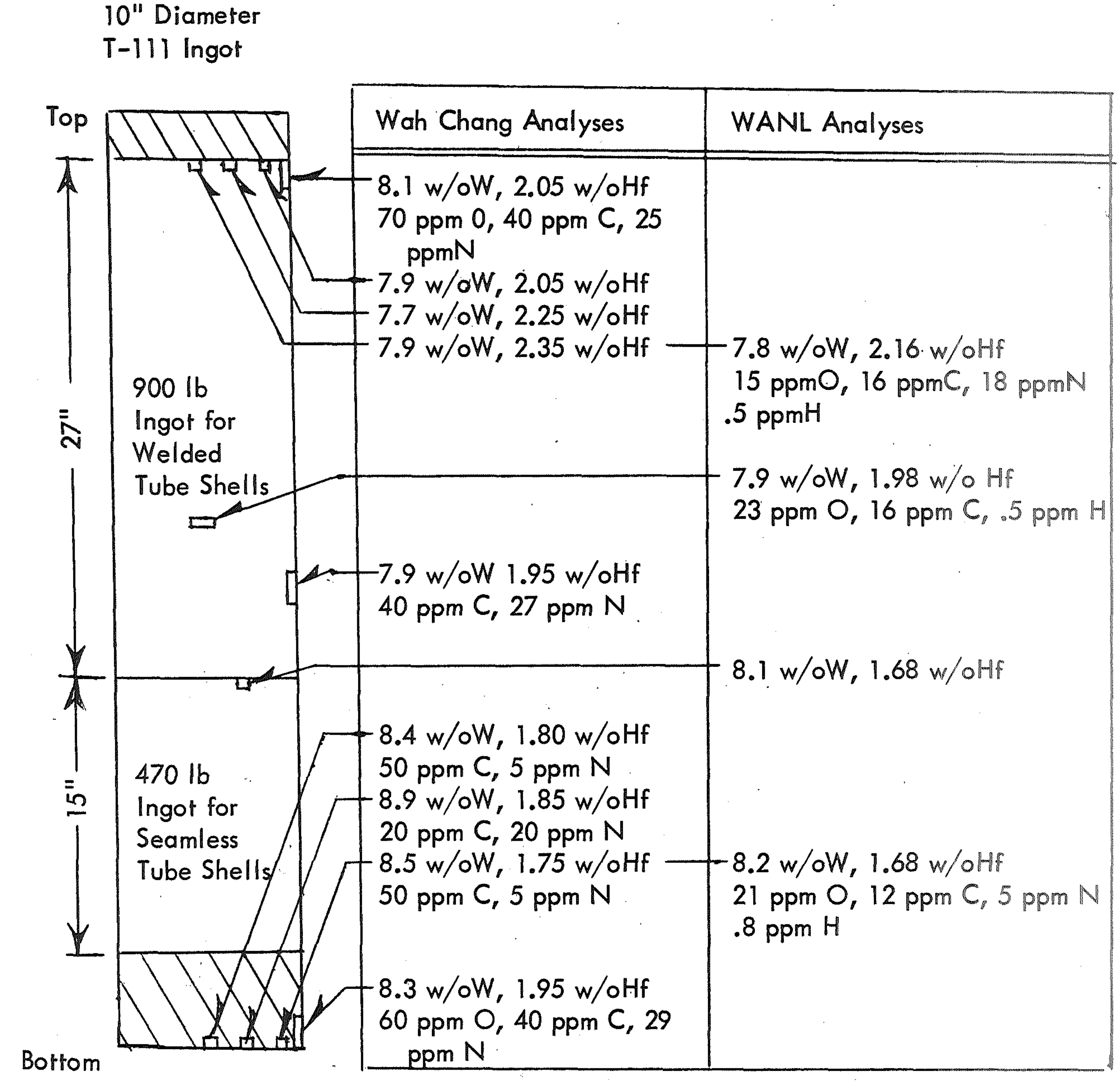


Figure 3-1. Ingot Melting Sequence



	Tungsten	Hafnium	Carbon	Oxygen	Nitrogen
Specification Limits	7-9 w/o (8 nom)	1.8-2.4 w/o (2 nom)	50 ppm max.	100 ppm max.	50 ppm max.

Figure 3-2. Location of T-111 Ingot Analyses

tabulated in Figure 3-2 verified that alloy additions were within specification and good homogeneity was achieved in the as-melted ingot. Trace impurity analytical results were also within specification and tabulated in Table 3-1. Hardness varied from 205 DPH at the ingot top to 210 at the bottom and grain size varied from ASTM -2 to -6 (1/32 to 1/8 inch dia.).

3.2 Seamless Tube Shell Extrusion

Billet Preparation

A 3 3/4 inch diameter core was removed from the 15 inch length of ingot by electro-discharge machining (EDM). The electro-discharge machining was performed by Thermo-Electron Corp.* A boring operation was used to remove the surface layer damaged during the electro-discharge machining process. The finished machined tube hollow-billet was 8.625 inch OD by 4.712 inch ID by 14.940 inch long and weighed 380 lbs. The machined finish on the OD and ID was 32 RMS. Macro and microstructures of the trepanned ingot section are shown in Figure 3-3. The clean single phase as-cast microstructure is indicative of the low interstitial content of the as-melted ingot. The T-111 hollow extrusion billet was clad with mild steel to provide protection from the salt bath during heating and the atmosphere during extrusion. Details of the instrumented mild steel clad hollow extrusion billet are illustrated in Figure 3-4. Temperature of the billet during heating was monitored by means of a stainless steel clad chromel-alumel dual junction thermocouple embedded in the base of the extrusion billet. An all welded, strain accommodating feedthrough was developed which performed satisfactorily during the salt bath heating cycle. A leak tight container was an absolute necessity since a preliminary experiment had shown that T-111 reacted with the 2300°F salt to a depth of 0.05-0.1 inch after exposure for 4 hours.**

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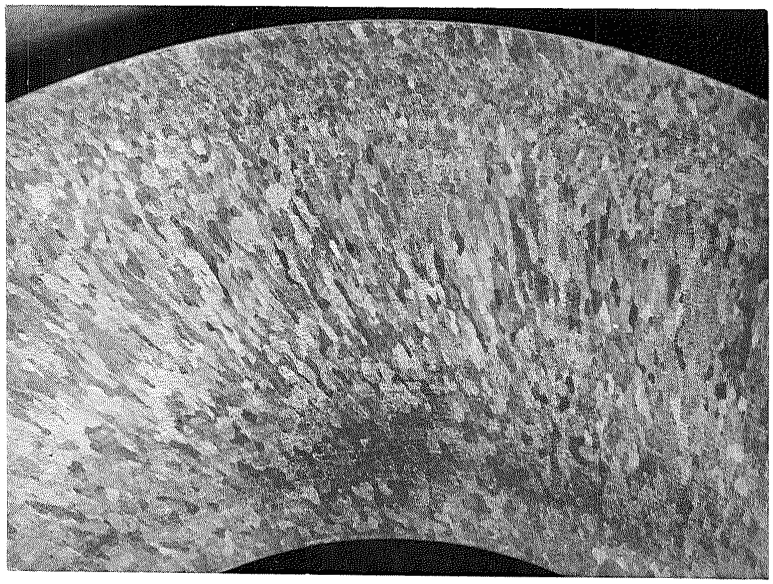
*See Appendix 1

**See Appendix 2.2

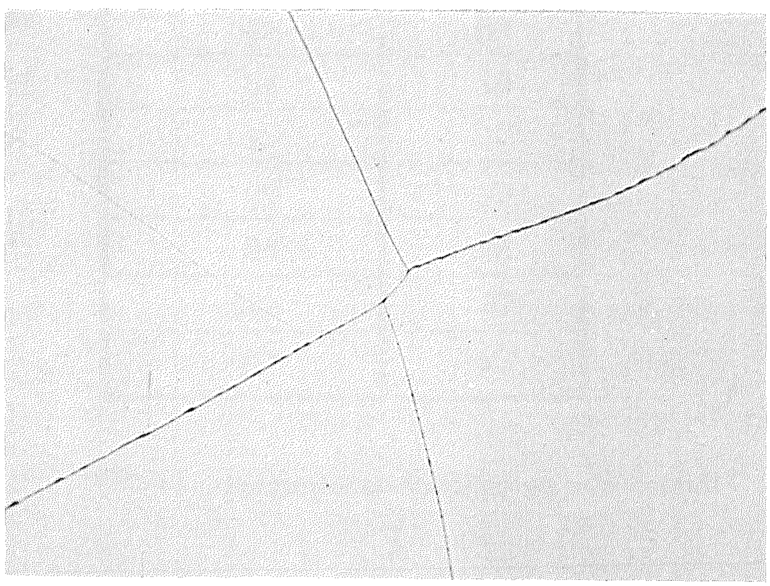
Table 3-1. Trace Impurity Analysis of T-111 Ingot*

Element	ppm by weight
Si	<60
Fe	<60
Cr	<60
Cb	1000
Mn	40
Ni	<60
Al	40
V	<20
Cu	20
Mo	60
Ti	<20
Co	<60

*Determined by emission spectroscopy.



a. Macrostructure 1.1X



17,250 200X

Figure 3-3. Trepanned Ingot Wall Section

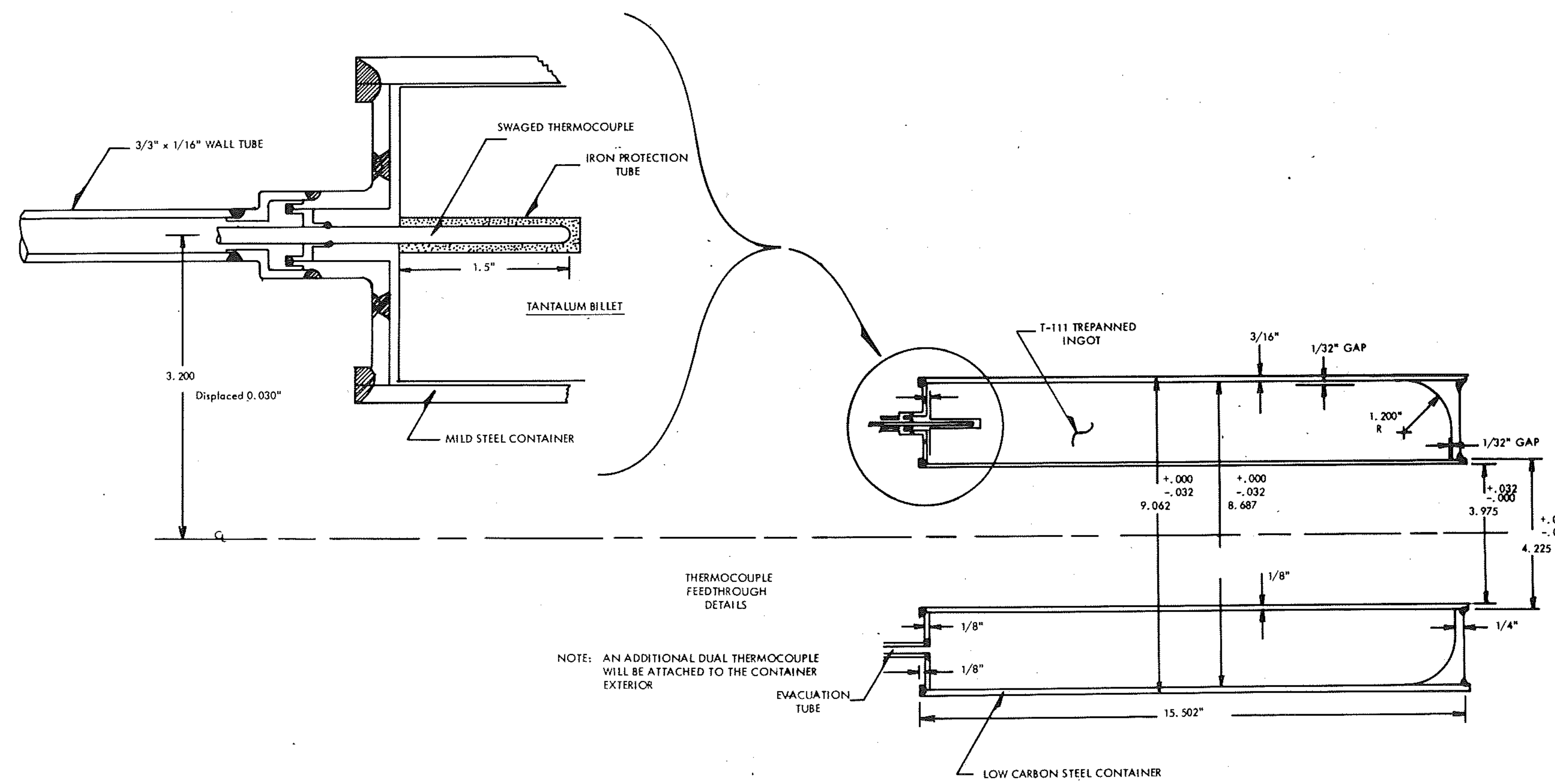


Figure 3-4. Tube Hollow Extrusion Design Drawing

The OD and ID surface of the T-111 billet was protected with tantalum foil to minimize any reaction with the mild steel container.* Shown in Figures 3-5 and 3-6 are the tantalum wrapped billet and mild steel container components. Except for a small tube, used as an evacuation and sealing vent, the mild clad container was assembled and sealed by inert gas-tungsten arc (GTA) welding.

After helium leak checking the welded joints to assure a leak free assembly, the clad billet was evacuated through the vent tube and heated to 200°F to outgas the internal surfaces. The clad billet received an overnight evacuation and was sealed by electron beam welding. The chamber pressure during EB seal welding of the clad billet was $< 5 \times 10^{-5}$ torr.

Seamless Billet Extrusion

The 9 1/16 inch diameter clad hollow billet was heated to 2300°F and successfully extruded over a fixed mandrel with a resultant 50 inch long x 6 1/8 inch OD tubular extrusion (See Figure 3-7). Extrusion parameters are listed in Table 3-2 and details of the extrusion press are in Appendix 1.

Heating of the clad billet was accomplished by immersion in a 2300°F neutral, barium chloride salt bath. The billet reached the salt bath temperature within 25 minutes and was soaked at 2300°F for an additional 60 minutes prior to extrusion. A total transfer time of 150 seconds was required after removal from the salt bath until the extrusion stroke was initiated. Although the nominal transfer time is 90 seconds, a tight fitting mandrel required an additional 60 seconds for final alignment of the billet.

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*See Appendix 2.3

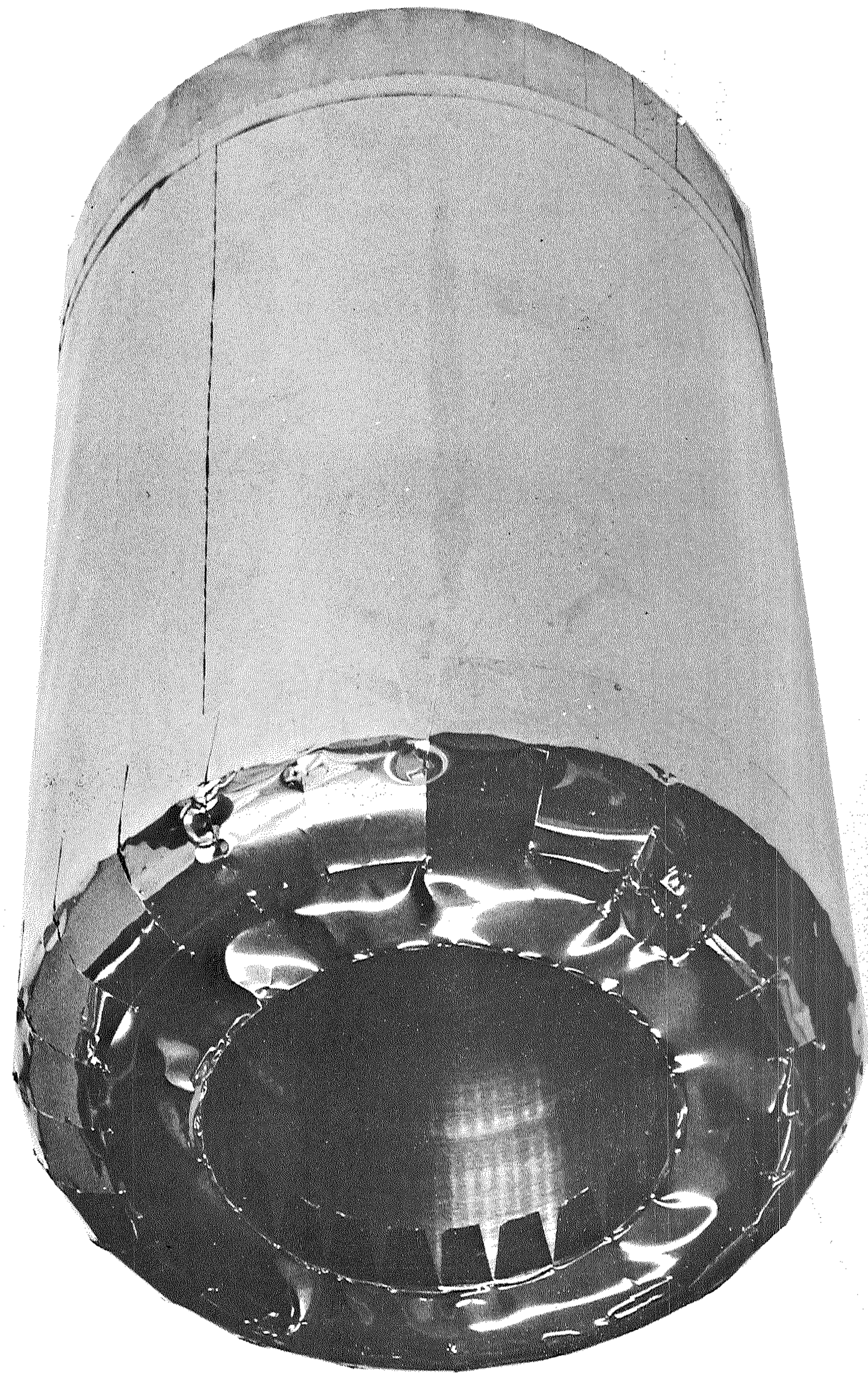

Figure 3-5. Tantalum Foil Wrapped Hollow Billet

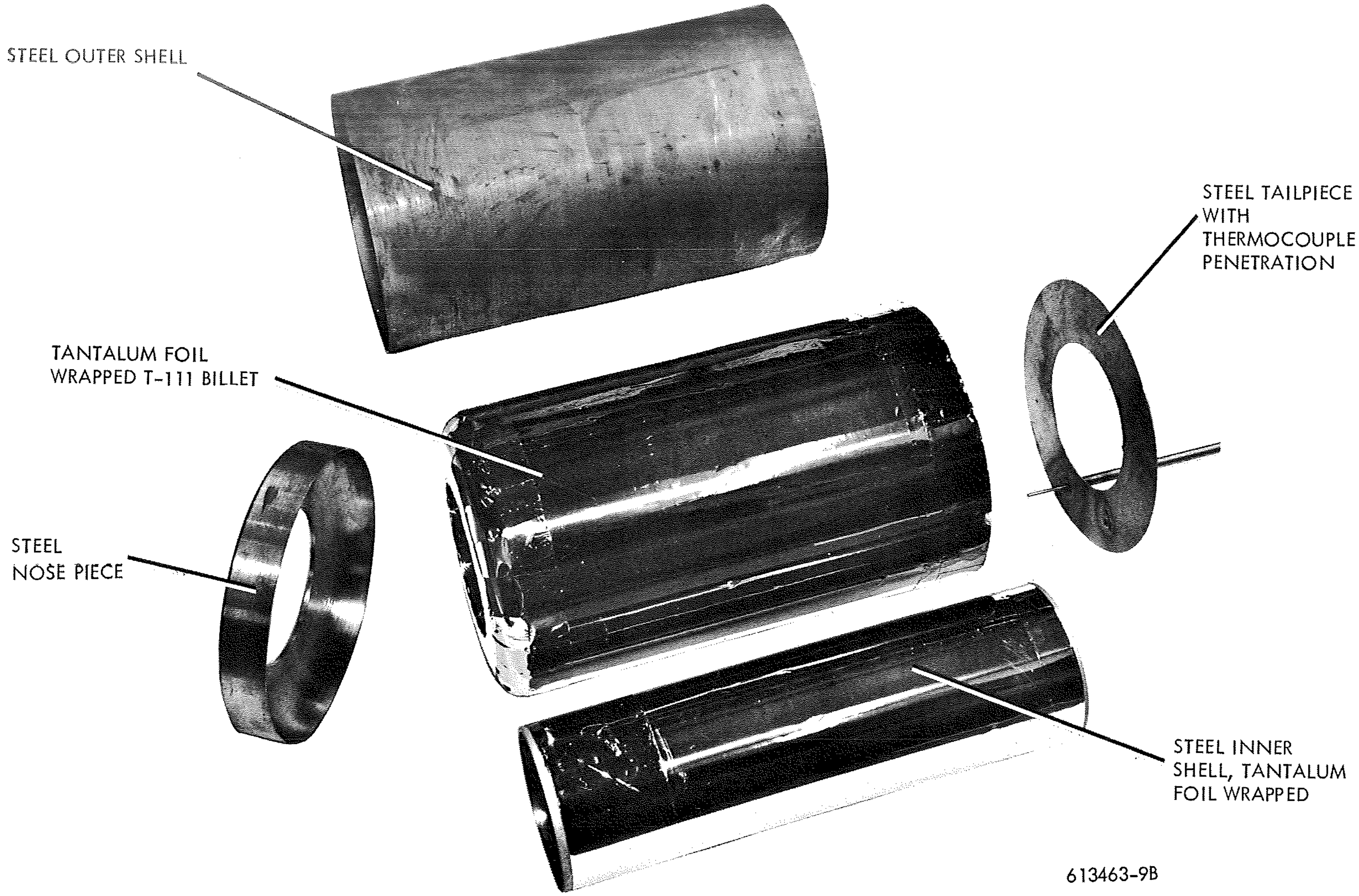

Figure 3-6. Hollow Extrusion Components

Figure 3-7. As Extruded Seamless Tube Shell with End Sections Removed

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Table 3-2. Tube Hollow Extrusion Results

Breakthrough Load	2, 790 tons (K = 42.7 tons/in^2)*
Running Load	2,710 tons (K = 41.5 tons/in^2)*
Ram Speed	2-3 inches/second
Reduction Ratio	3.4:1
Container Temperature	600°F
Container Diameter	9 1/4 inches
Mandrel Diameter	4.335 inches
Lubricant	Petroleum Base

*Calculated from

$K = F/A \ln R$

K = extruding constant, tons/in^2

F = ram force in tons

A = container cross-sectional area in^2

R = extrusion ratio $\left(\frac{\text{area of container}}{\text{area of die opening - area of mandrel}}\right)$

The mild steel cladding, which had remained intact during the heating and extrusion operation, was removed by pickling in a 50-50 nitric acid-water solution. The rough textured surface of the dejacketed extrusion shown in Figure 3-8 was not unexpected and is most likely caused by the large grain size of the as-cast ingot. Dimensional uniformity of the extruded tube shell was excellent as shown by the data in Table 3-3. The hollow extrusion was straight within 1/32 inch over the 50 inch length. A maximum ovality of 1/8 inch was measured midway along the length.

The extrusion was machined to a final size of 5.765 inch OD x 4.610 inch ID x 44.65 inch in length. The final wall thickness of 0.578 inches was greater than the target size of 0.375 inch because of the excellent dimensional uniformity of the extruded billet. A minimum of 0.050 inches was removed from each extruded surface to assure the removal of any contaminated surface layer formed during the hot extrusion operation.

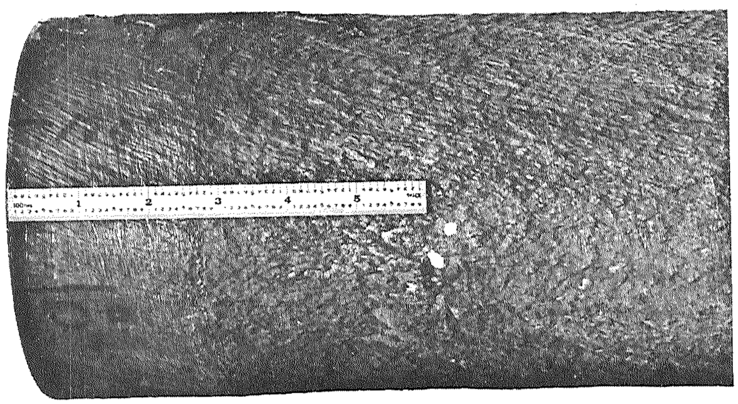
The machined tube shell was honed and polished to a surface finish of 32 RMS and is shown in Figure 3-9. Radiographic, dye penetrant, and ultrasonic inspection indicated that the tube shell was sound and free of defects.

The machined and polished tube shell was chemically cleaned by pickling in a solution of 60 volume percent water, 30 volume percent nitric acid and 10 volume percent hydrofluoric acid. A minimum of 0.002 inches was removed from each surface to ensure elimination of any contamination from the prior machining and honing operations. After visual examination of the as-pickled surfaces, the shell was thoroughly water rinsed and swab dried. The as-pickled tube shell was wrapped with 2 layers of clean 0.002 inch tantalum foil to further minimize contamination during the vacuum annealing operation. Annealing was performed for 1 hour at 3000°F at $< 5 \times 10^{-5}$ torr which resulted in a decrease in hardness as well as refinement of the grain size as illustrated by the microstructures shown in Figure 3-10. Oxygen analysis

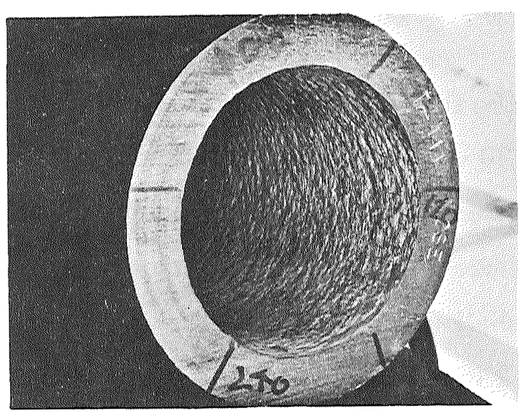
Rear Front

Bare T-111 Extrusion

Front



Exterior Surface



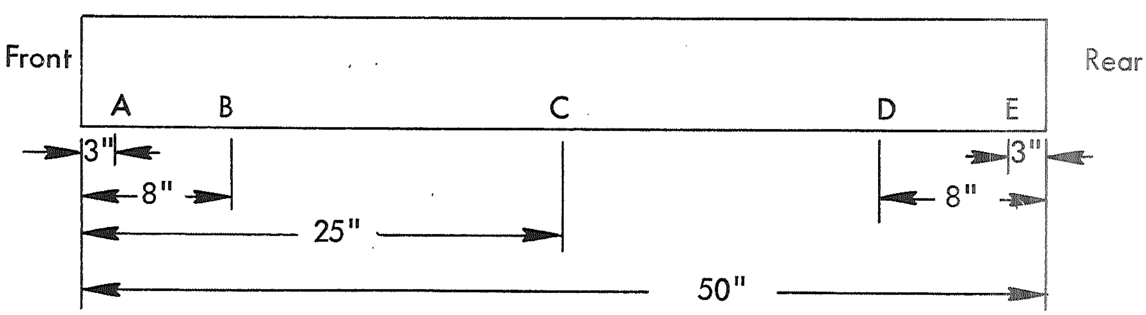
Interior Surface (Front)

Figure 3-8. Surface Appearance of Extruded Seamless Tube Shell with Steel Container Removed

Table 3-3. Extruded Seamless Tube Shell Dimensions after Steel Cladding Removal

		Location				
Position	Dimension	A	B	C	D	E
0°	O.D.		5.943	5.955	5.951	
	I.D.		4.393	4.402	4.430	
	Wall	0.842	0.778		0.786	0.811
120°	O.D.		5.915	5.892	5.961	
	I.D.		4.382	4.406	4.412	
	Wall	0.854	0.776		0.798	0.823
240°	O.D.		5.987	6.013	5.977	
	I.D.		4.462	4.435	4.412	
	Wall	0.849	0.788		0.793	0.802

Overal extrusion length after cropping = 50"
Camber = 1/32"
Maximum ovality at position C = 0.121"



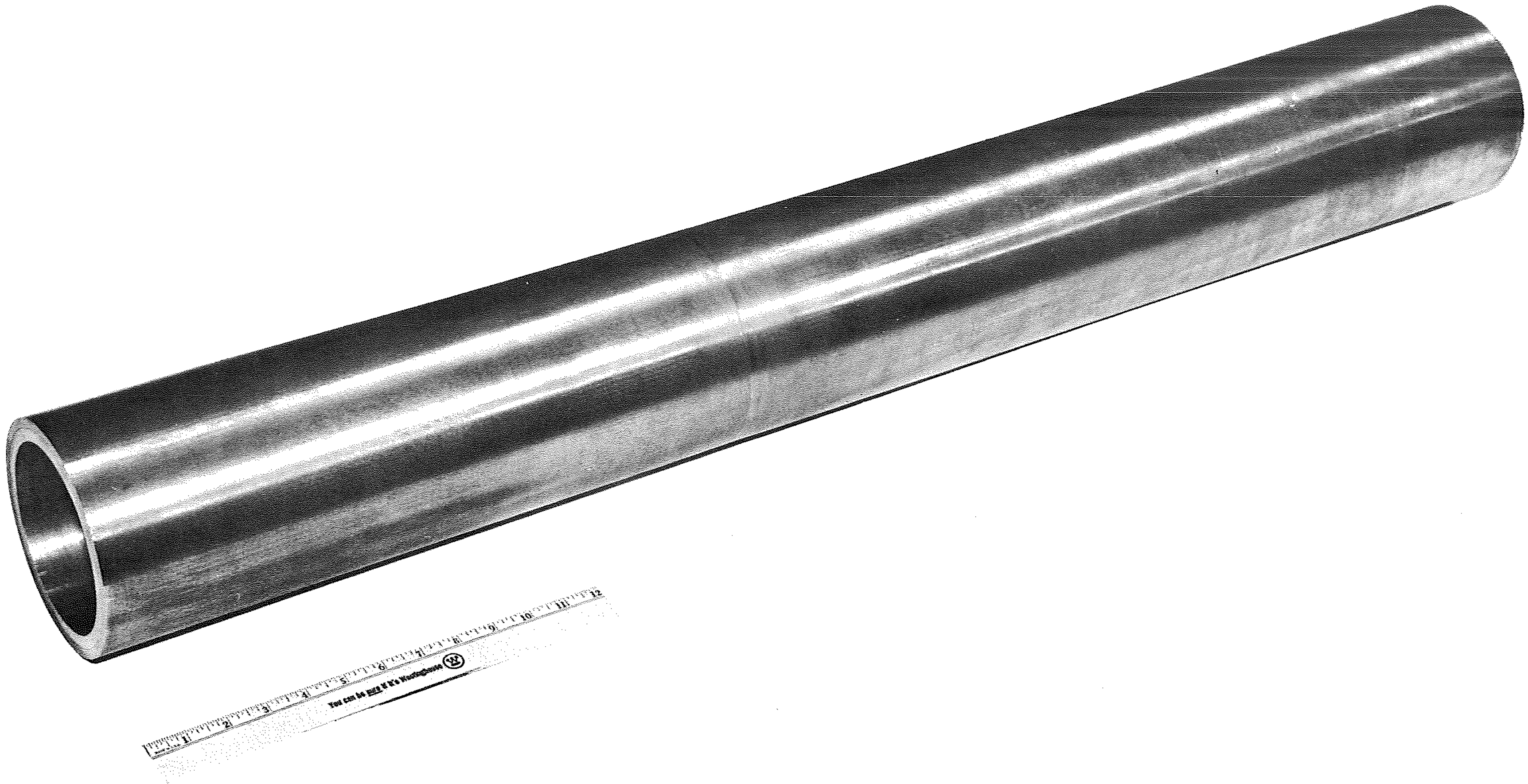
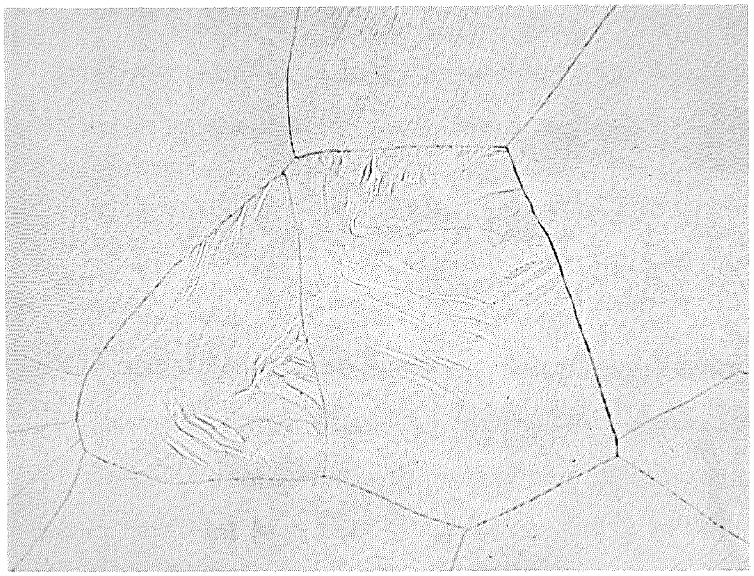
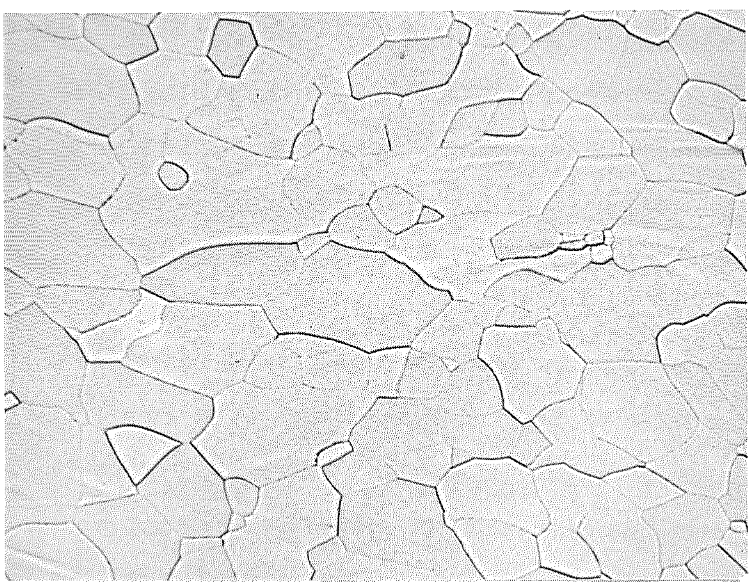


Figure 3-9. Finish Machined Seamless Tube Shell



As Extruded Hardness
266 DPH

19,281 100X



ASTM Grain Size 4 to 5

Annealed Hardness
204 DPH

20,578B 100X

Figure 3-10. Transverse Sections of Seamless Tube Shell Extrusion As Extruded and after Annealing 1 hour at 3000°F of the annealed tube shell was identical to the as-cast analysis, 25 ppm vs 24 ppm, verifying the adequacy of the processing controls. A more detailed description of the vacuum annealing furnace operation is in Appendix 1.

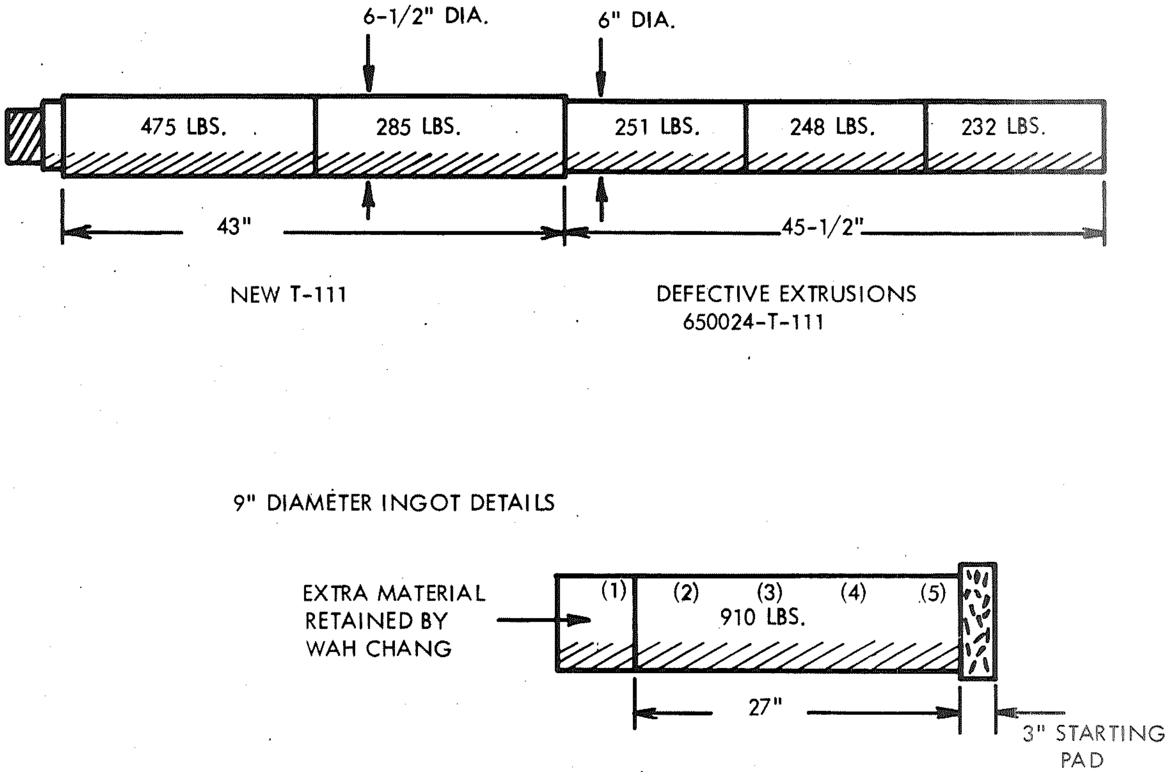
4. WELDED TUBE SHELL PROCESSING

The welded tube shell was produced by press forming and longitudinal welding cylinders of .4 inch thick T-111 plate. The plate was produced from a 9 inch diameter ingot by an initial hot breakdown of the ingot to 5 inch diameter rounds, hot forging the rounds to 1 1/4 inch thick sheet bar, and cross rolling the sheet bar to 3/8 inch thick, 20 inch x 24 inch flat plate.

4.1 Welded Tube Shell Ingot Melting

Originally it was intended to produce all the material for this program from a single large T-111 ingot. Initial extrusion difficulties described in Appendix 2 with the welded tube shell ingot required remelting and the addition of approximately 30% of new T-111 melting stock. Throughout the program, no significant differences between the seamless and welded tubing were observed which could be attributed to ingot history so the original intent was satisfied. Details of the defective extrusion and corrective action are presented in Appendix 2.

The electrode configuration and chemical analyses of the ingot are shown in Figure 4-1. The remelted ingot was cast into a 9 5/8 inch diameter mold and at a cast length of 40 inches weighed 1500 lbs. Consumable arc melting was accomplished at from 1 to 5 microns pressure and 17,000 amperes and 38 volts (650 KW) completed the melt in slightly over 1 hour. A stirring coil was used during the melt with a direction reversal every 2 seconds. The as-cast grain size was similar to the originally melted ingot and a complete macrosection of the ingot top is shown in Figure 4-2. Photomicrographs of the ingot top are shown in Figure 4-3. The greater amount of substructure evident in Figure 4-3 as compared to Figure 3-3 most likely reflects the difference in cooling rate since this sample was obtained from the top of the ingot. Average hardness values were 213 DPH for the remelted ingot as compared to 208 DPH for the original.



INGOT ANALYSES LOCATION NOTED ABOVE
AS FURNISHED BY VENDOR

ELEMENT		POSITION - % PPM 1	2	3	4	5	AVERAGE OF WESTINGHOUSE CHECK ANALYSIS
W	w/o	8.1	8.4	8.0	7.8	7.8	8.6
Hf	w/o	2.0	1.9	2.0	1.9	2.0	2.0
C	ppm	<20	<20	<20	20	25	30
N	ppm	14	9	7	12	10	10
Fe	ppm	20	20	<20	<20	20	50
Mo	ppm	15	15	<10	10	15	20
Cb	ppm	290	290	340	440	415	--
O	ppm	60	--	--	--	30	22

Figure 4-1. Welded Tube Shell Ingot Preparation and Chemical Analysis

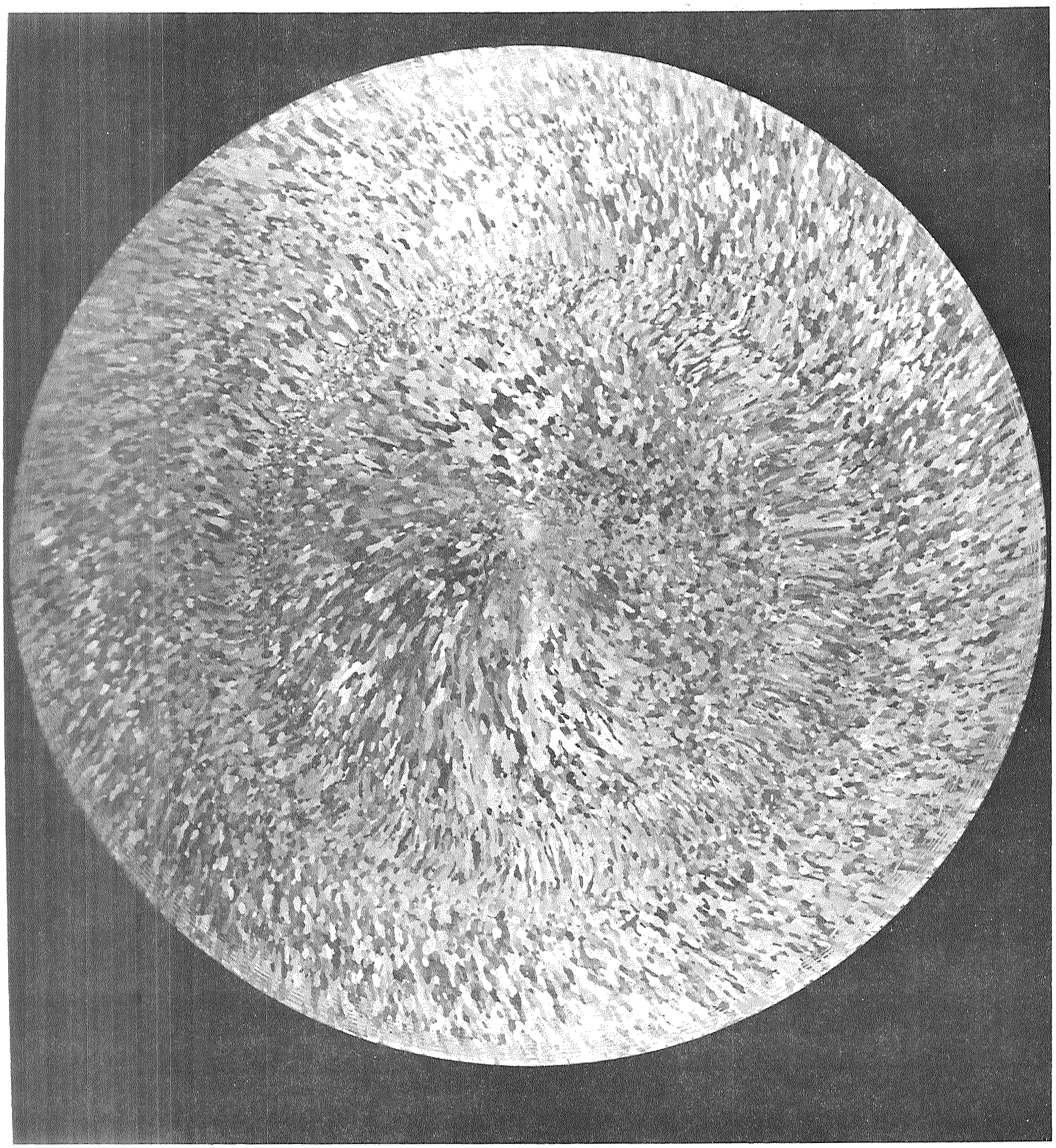
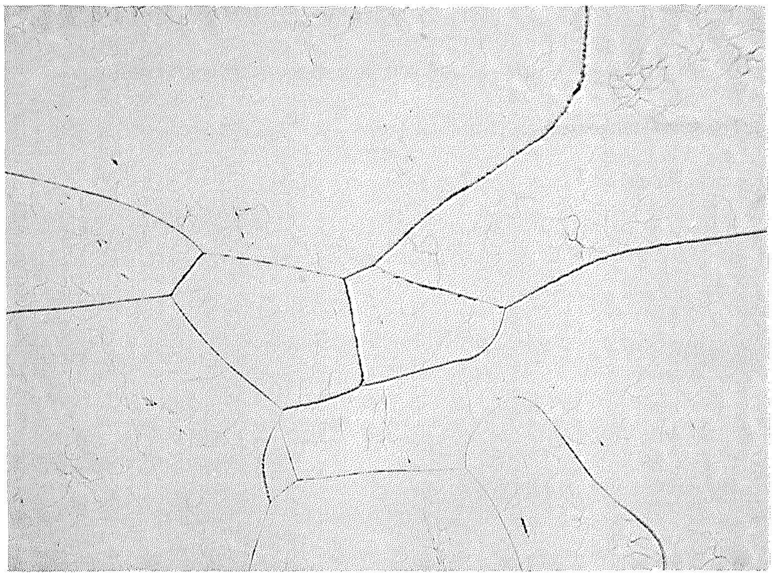
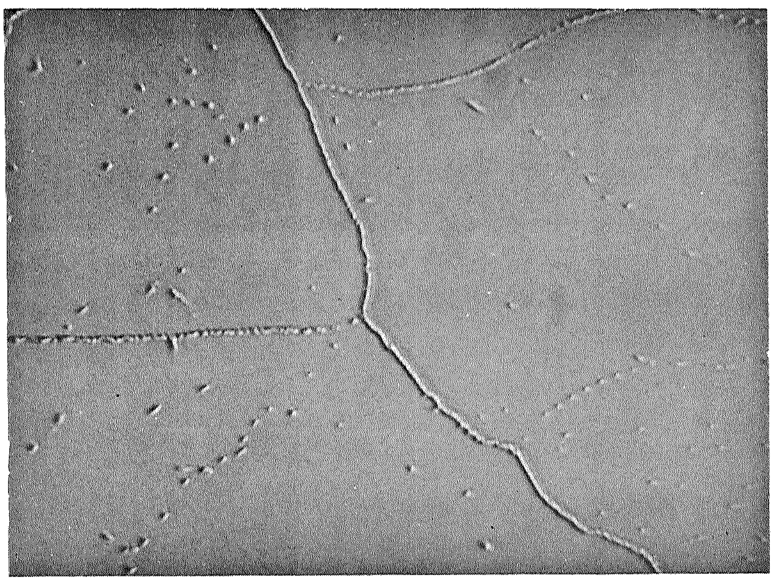


Figure 4-2. 9 Inch Diameter T-111 Ingot Top



18,369 50X



18,370 1500X

Figure 4-3. Section from Top of Remelted T-111 Ingot for Welded Tube Shell

Contact ultrasonic inspection of the conditioned ingot indicated soundness with no internal porosity or pipe. Liquid penetrant inspection of the ingot slice shown in Figure 4-2 showed no porosity. The complete chemical analyses and grain size information is listed in Sections 6.3 and 6.1 respectively.

4.2 Extrusion for Welded Tube Shell Material

The initial breakdown for the welded tube blank material was a 3.3:1 2300°F extrusion from an 8.44 inch diameter ingot to a 5 1/4 inch diameter extruded round.

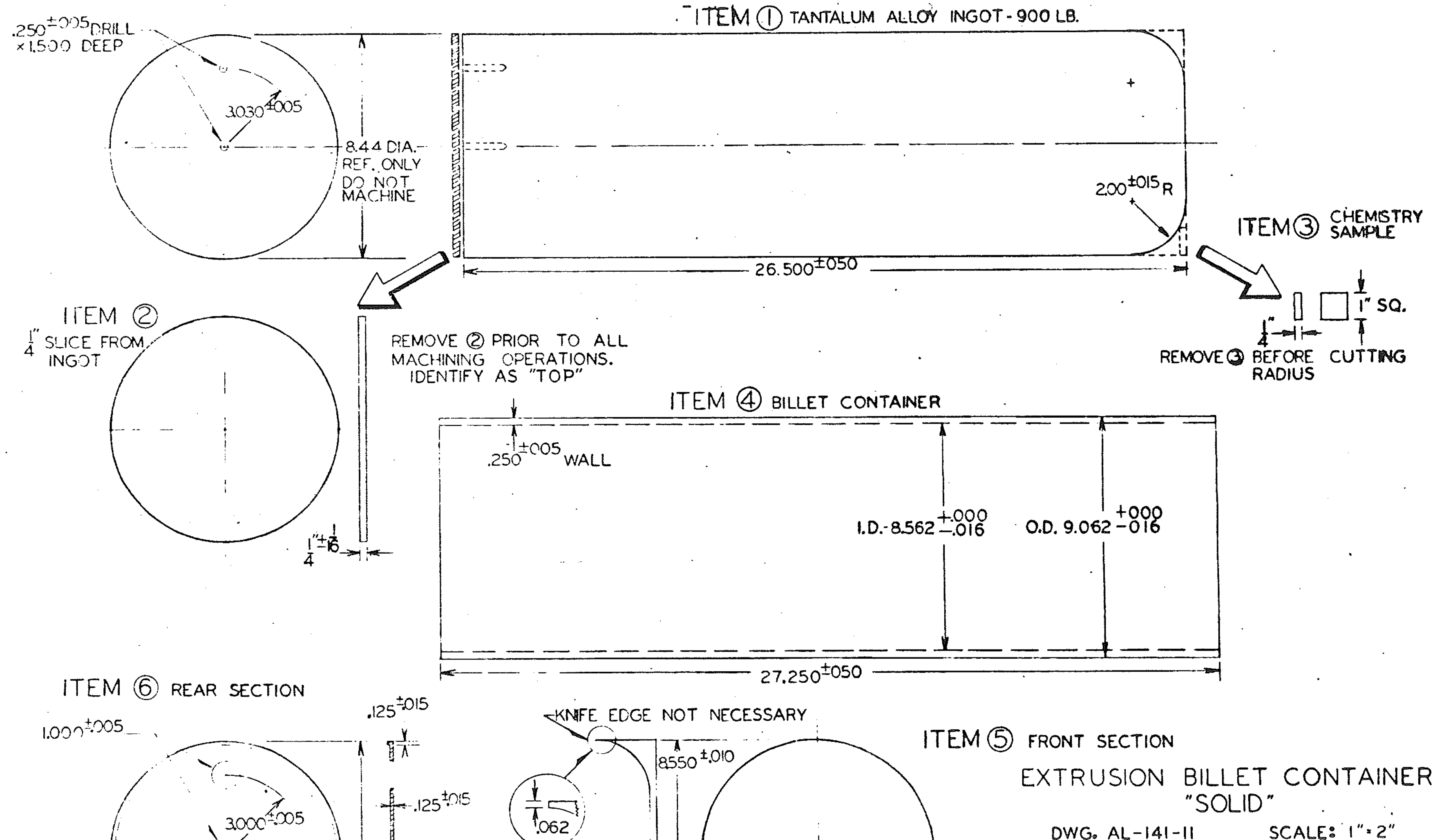
Extrusion Billet Preparation

The same procedure used for encapsulating and instrumenting the tube hollow billet was used for the solid billet. Details of the solid billet protective container are shown in Figure 4-4. The tantalum foil wrapped billet and assembled protective container are shown in Figure 4-5. Dual junction thermocouples were embedded 1 1/2 inches deep at the billet center and mid-radius positions. The thermocouple extension tubes shown in Figure 4-5 were bent flat for shipment. The billet was placed horizontally in the salt bath and the thermocouple extension tubes were straightened to the vertical position to clear the salt bath surface.

Extrusion for Welded Tube Shell Material

After immersion, the sealed billet reached the salt bath temperature of 2300°F in 70 minutes, and the billet was soaked for an additional 60 minutes prior to extrusion. Figure 4-6 compares the heating rates of the solid billet and the hollow billet described in Section 3.3. On removal from the salt bath, the ends of the steel container were concave due to atmospheric and salt bath pressure verifying no leakage occurred. The billet was transferred to the press within 90 seconds and extruded in 15 seconds. The extrusion results are tabulated in Table 4-1, and the extrusion constant (K) was similar to that for the hollow extrusion (See Section 3.3).

The 5 1/4 inch diameter extrusion was 84 inches long with approximately 1/2 inch camber near the front section. The steel container was intact following extrusion with no evidence of



ITEM NO.	DESCRIPTION	MATERIAL
1	900 LBS. TANTALUM INGOT	TANTALUM (T-111)
2	1/4" SLICE FROM INGOT TOP	TANTALUM (T-111)
3	CHEMISTRY SAMPLE FROM INGOT BOTTOM	TANTALUM (T-111)
4	BILLET CONTAINER	1010 STEEL[a]
5	FRONT SECTION	1010 STEEL
6	REAR SECTION	1010 STEEL

(a) ORDER SEAMLESS MECHANICAL TUBING 9-1/4" OD × 1/2" WALL

Figure 4-4. Solid Extrusion Billet Container

1 Layer of 0. 0025" Tantalum Foil

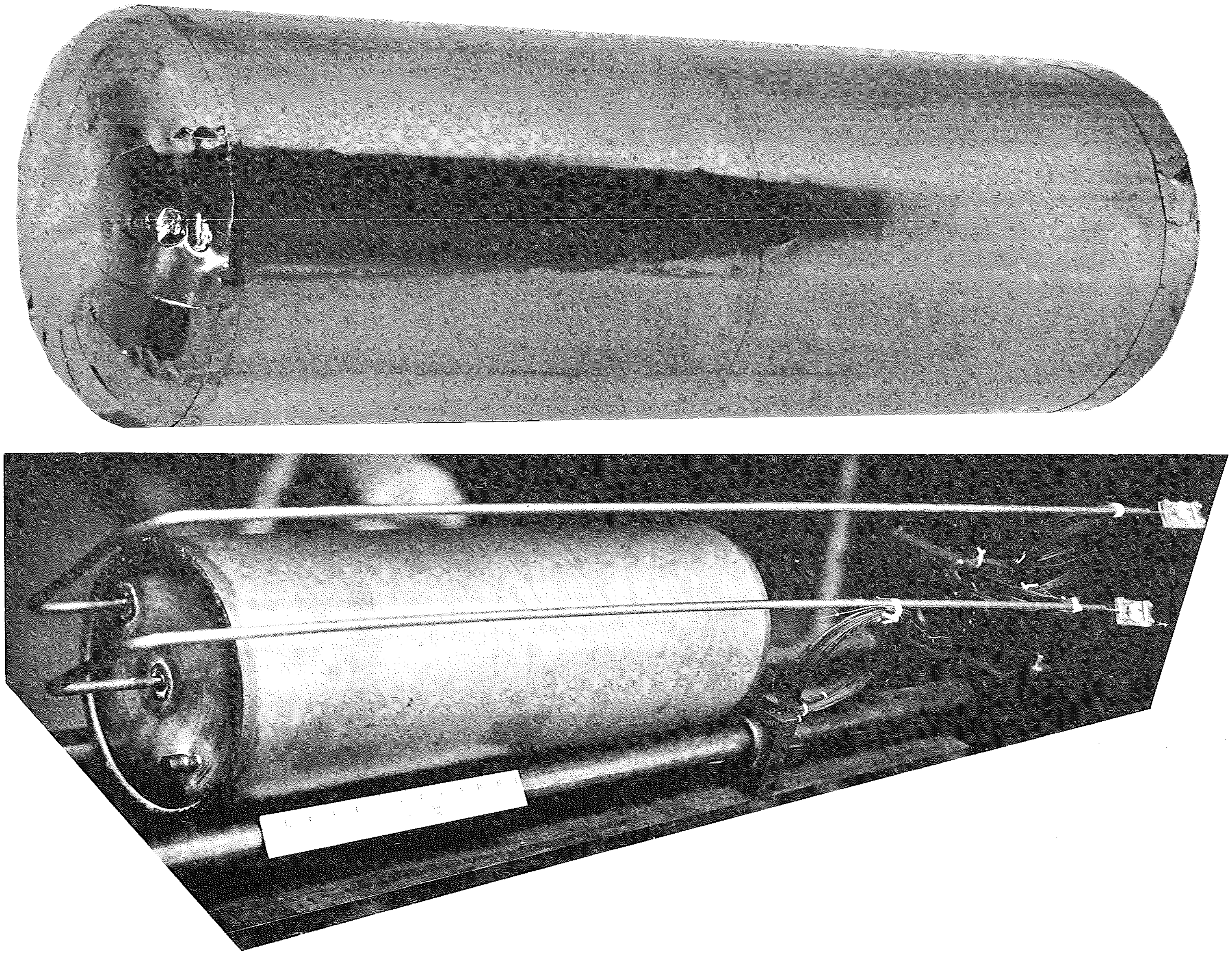


Figure 4-5. Solid Extrusion Billet

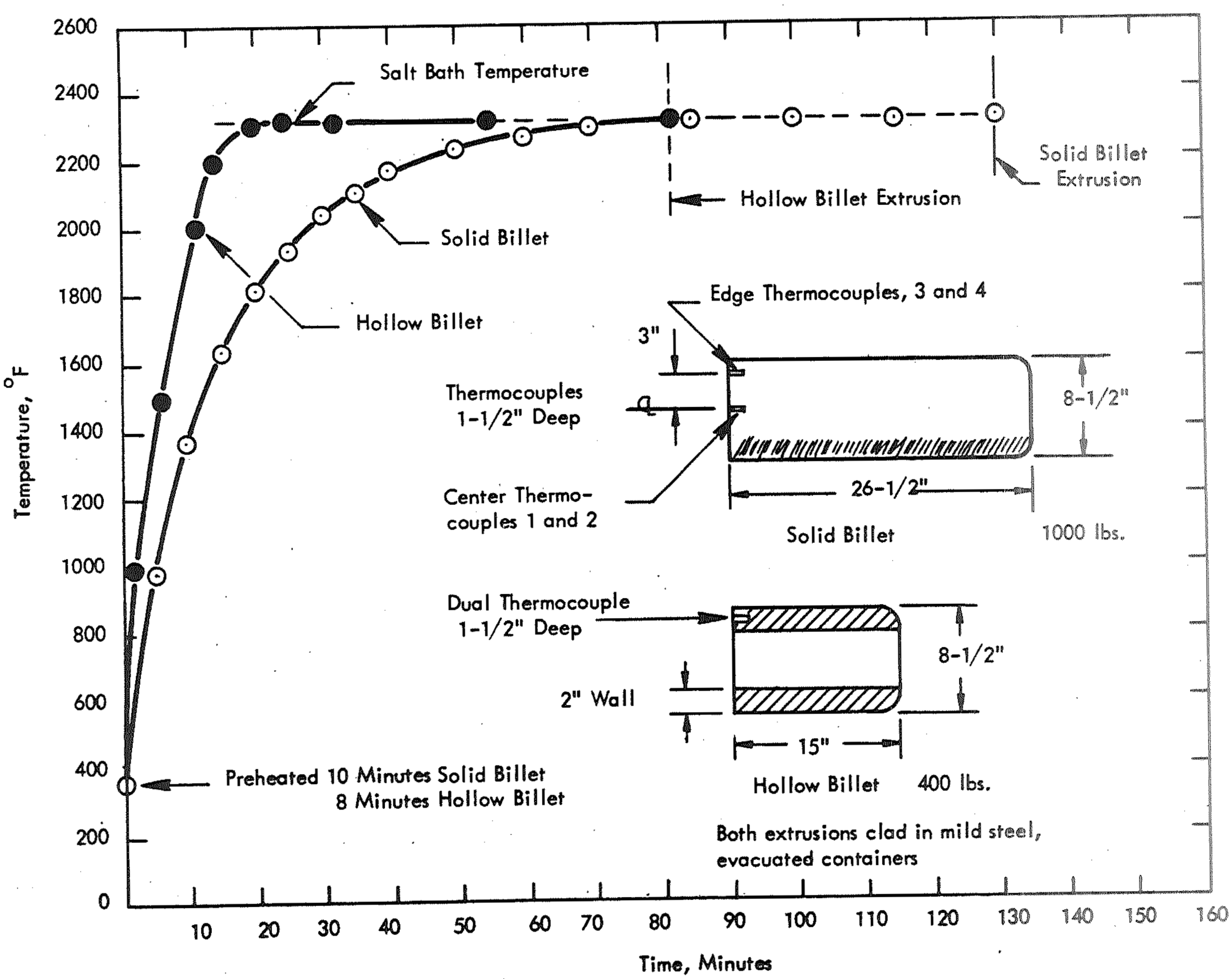


Figure 4-6. Salt Bath Heating Rates of Solid and Hollow Extrusion

Table 4-1. Solid Billet Extrusion Results

Breakthrough Load	3,270 tons (K* = 40.7 tons/in^2)
Running Load	3,190 tons (K* = 39.8 tons/in^2)
Ram Speed	2-3 inches/second
Reduction Ratio	3.3:1
Container Temperature	600°F
Container Diameter	9 1/4 inches
Lubricant	Petroleum Base

*Calculated by $K = F/A \ln R$

K = extrusion constant, tons/in^2

F = ram force in tons

A = container cross-sectioned area, in^2

R = reduction ratio $\left(\frac{\text{area of container}}{\text{area of die opening}}\right)$

container rupture during salt bath heating or during extrusion. The extrusion was sectioned and the machined extrusion rounds were 4 1/2 inch OD by 23 inches long and weighed approximately 230 lbs. each. Ultrasonic and liquid penetrant inspection showed the billets to be sound and free of defects.

Vacuum Annealing

The billets, conditioned by lathe turning and grinding, were pickled, cleaned and wrapped in tantalum foil prior to vacuum annealing for 1 hour at 3000°F and $< 5 \times 10^{-5}$ torr. As previously discussed, a minimum of 0.002 inches of each surface was removed by pickling to ensure removal of any contaminated layer produced during machining.

The 1 hour at 3000°F annealing treatment resulted in a reduction in hardness and refinement of the grain structure as shown in Figure 4-7.

4.3 Sheet Bar Forging for Welded Tube Shell

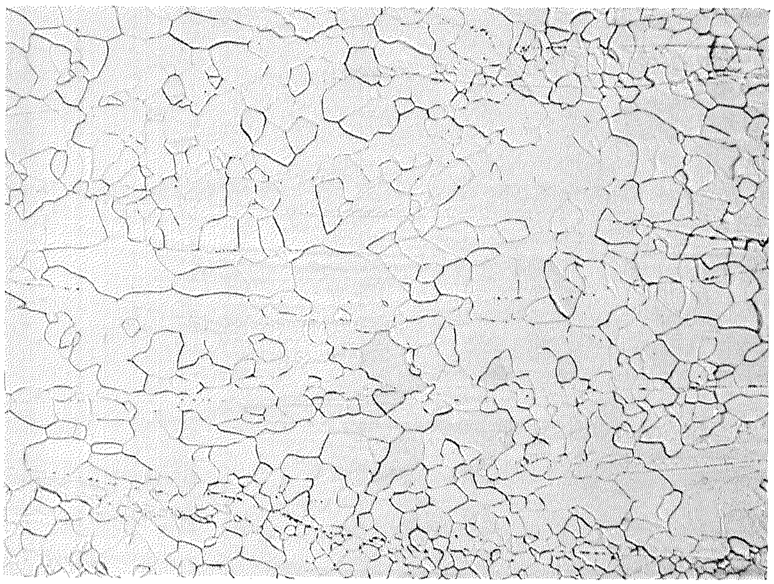
The annealed extrusion billets were forged to sheet bar and cross rolled to produce .4 inch plate for the tube forming operation as shown in Figure 4-8.

Aluminum - 12 w/o Silicon Protective Coating

To prevent excessive scaling loss during the hot forging operation at 2300°F, the 4 1/4 inch diameter x 23 inch long extruded rounds were coated with an Al-12 w/o Si alloy by immersion. Tantalum alloy hanger straps were attached as shown in Figure 4-9. The 230 lb. billets were immersed vertically in a large ladle of aluminum alloy covered with a layer of flux of 46 w/o KCl, 35 w/o NaCl, 13 w/o $NaAlF_3$ and 6 w/o AlF_3. The flux assured wetting of the T-111 billet by the aluminum alloy. The molten aluminum alloy was maintained at 1700°F to insure a good adherence of the protective coating.

As Extruded Front Section

18,796 261 DPH 50X



Vacuum Annealed
1 hr. 3000°F
Center Section

18,801 217 DPH 50X

Figure 4-7. Longitudinal Sections of Extrusion Comparing As-Extruded to Annealed Structure

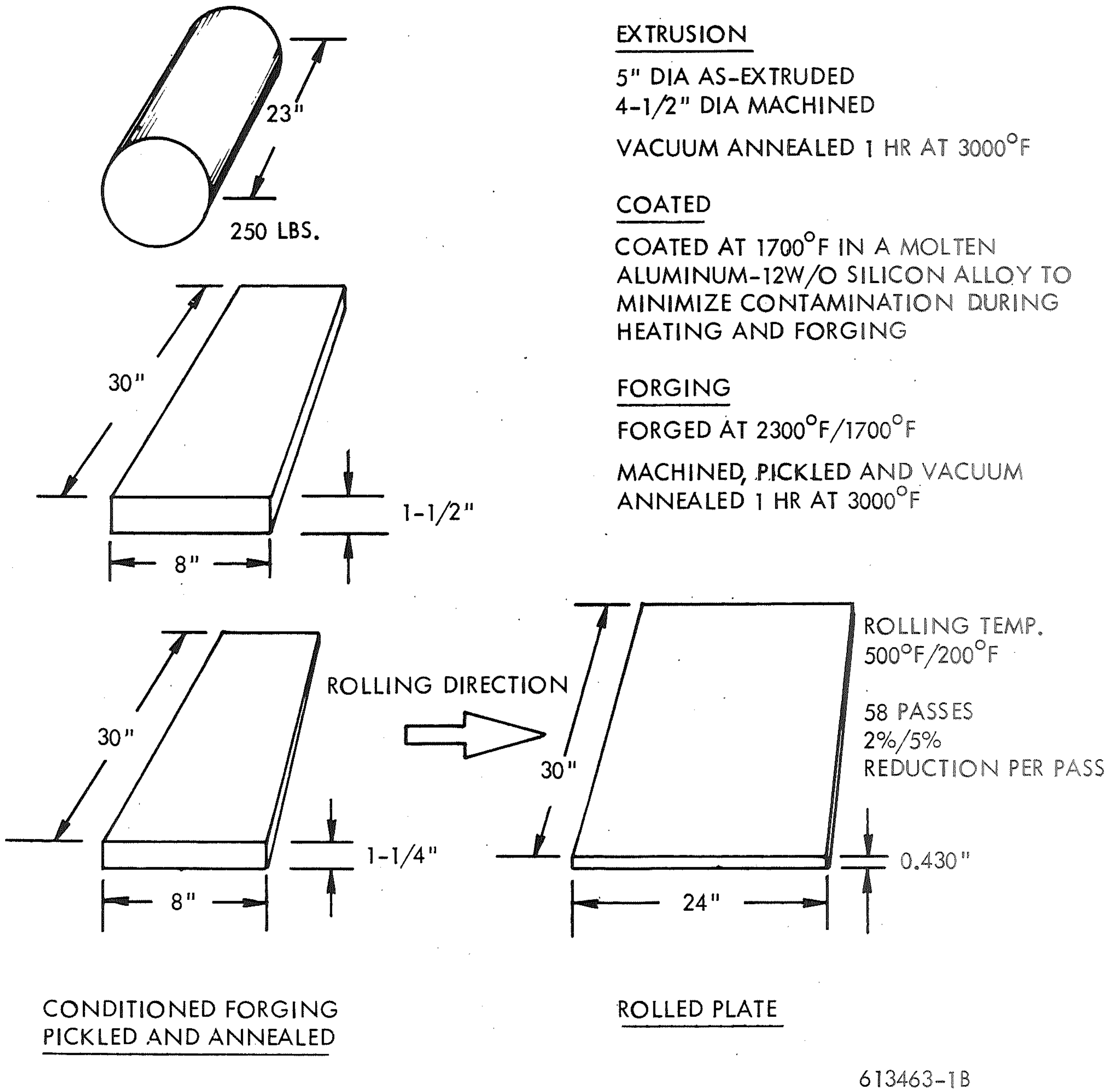


Figure 4-8. Processing Sequence for Conversion of T-111 Extrusion to Plate

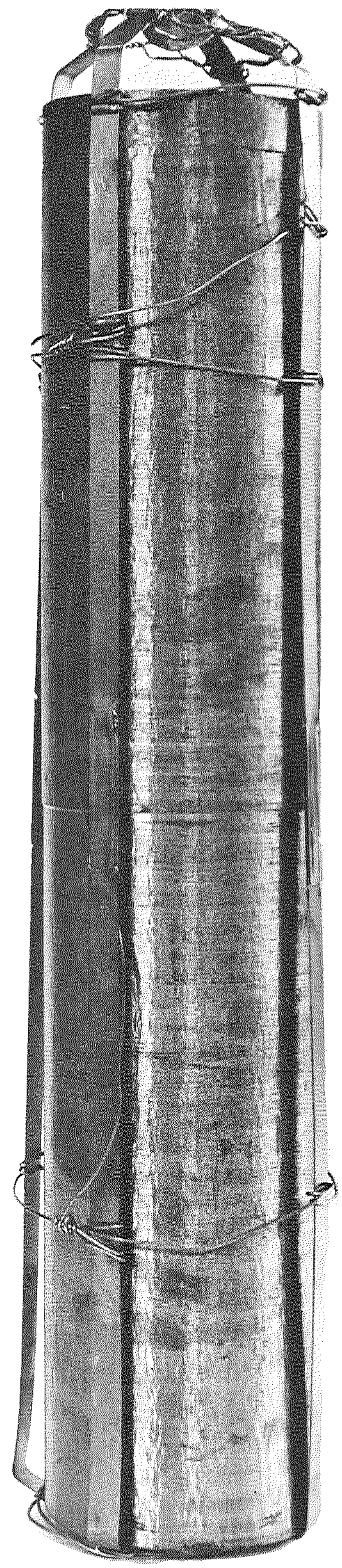


Figure 4-9. T-111 Billet Prepared for Coating

A note interesting to future processing is that molybdenum wire used to reinforce the tantalum alloy hanger straps was rapidly dissolved by the aluminum alloy at temperatures over 1700°F.

Forging

The Al-12 w/o Si coated billets were hammer forged at the Industrial Forge Corporation*. A 20,000 lb. steam hammer was used for initial working and final forging was done on a 10,000 lb. hammer. The billets were heated in a gas fired furnace to 2350°F to 2400°F and forged until the temperature dropped to 1700°F. From 4 to 6 reheats were required on each billet to obtain the finished size. The forging schedule shown in Table 4-2 was typical.

The billet width and length during the initial forging reductions were controlled with a spreader bar. Temperatures were measured using an optical pyrometer. All three forgings were visually sound. Subsequent ultrasonic and liquid penetrant inspection indicated the condtioned bars to be free of defects. The conditioned sheet bar forgings (See Figure 4-10) were cleaned, pickled, wrapped in tantalum foil and then vacuum annealed for 1 hour at 3000°F at $< 5 \times 10^{-5}$ torr.

The recovery of sound material from the coated billets was better than 80% (See Table 4-3). This high yield is attributed to the use of the Al-12% Si coating.

The forging and subsequent annealing operation resulted in a further grain size refinement. The as-recyrstallized grain size of the forged plate was ASTM 7-8 and is shown in Figure 4-11 along with the as-forged microstructure.

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*See Appendix 1.

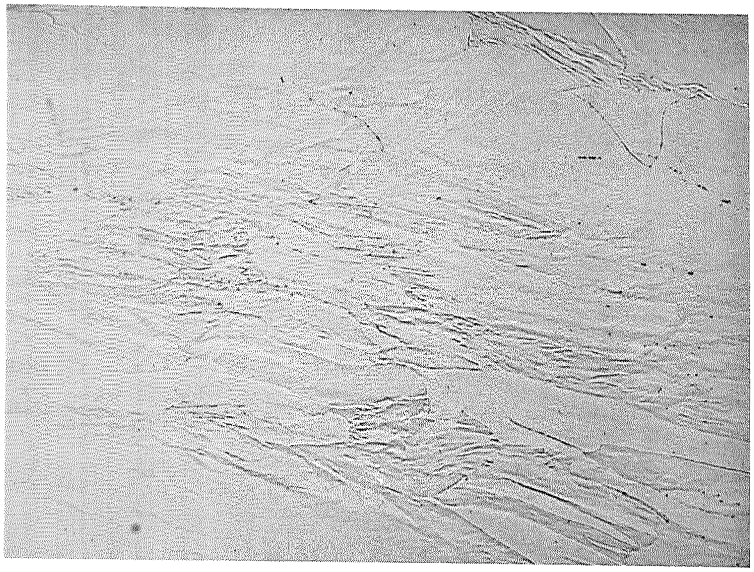
Table 4-2. Forging Schedule for T-111 Billet "C"

In	Out	Starting Temp. °F	Finishing Temp. °F	Size
10:40	11:10	2400	1900	
11:15	11:25	2400	1900	8 3/4 x 1 3/4 x 24
11:30	11:35	2250	1750	8 3/4 x 1 5/8 x 26
11:40	11:45	2250	1750	
11:50	11:55	2200	1400	8 3/4 x 1 5/16 x 30 1/2

Table 4-3. Forging Billet Yield

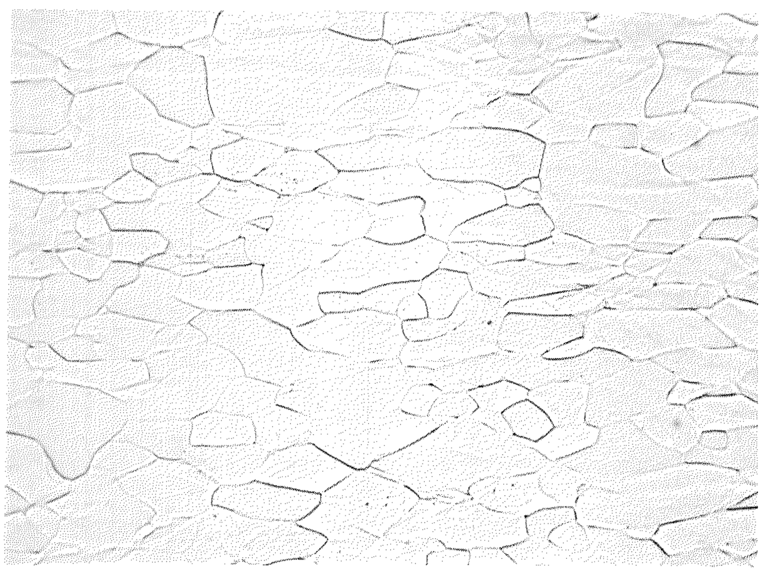
Billet	Starting Weight Pounds	Final Conditioned Weight, Pounds	Recovery, %
A	227	178	82
B	237	184	78
C	235	198	84

Figure 4-10. Forged Sheet Bar After Conditioning by Grinding to Remove a Minimum of 1/16 Inch Per Surface



18,994 200X

As Forged



19,390 200X

Annealed 1 hr. at 3000°F
ASTM G.S. 7-8

Figure 4-11. Comparison of As-Forged And Annealed Microstructure of Sheet Bar A

4.4 Plate Rolling

Two of the 1 1/4 inch thick, 30 inch long sheet bars (Bars B and C) were cross rolled in the 8 inch width direction to 24 inches x 30 inches x .430 inches. The rolling was accomplished at the Stellite Div., Cabot Corporation* on a 1200 HP Schloemann two high 52 inch wide mill with 32 1/2 inch diameter rolls. Sheet bar, A, was held in reserve. Approximately 60 rolling passes at 5% to 2% reduction ratio each were required to reach final size at a rolling temperature of from 500°F to 200°F. The mill reduction per pass was adjusted to the maximum mill separating force which ranged from 1500 to 1600 tons.

The anticipated rolling schedule assumed 10% or greater reduction per rolling pass with an initial rolling temperature of 800°F being maintained by the rolling deformation. In practice, however, only light reductions could be accomplished and the rolling temperature was slightly lower than desired. The as-rolled plate was cleaned, pickled, wrapped in foil and annealed 1 hour at 3000°F and $\leq 5 \times 10^{-5}$ torr (See Figure 4-12). Metallographic sections of the rolled and annealed plate are shown in Figure 4-13.

The as-recrystallized grain size of ASTM7-8 for the rolled plate was identical to that of annealed forged sheet bar. Thus no further grain refinement resulted from the rolling operation. A hardness traverse from the center to the edge of the rolled plate varied from 313 to 322 DPH indicating the uniformity of the worked structure.

The absence of any significant contamination during the prior hot working and annealing operation is verified by the interstitial analytical results in Table 4-4.

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*See Appendix 1

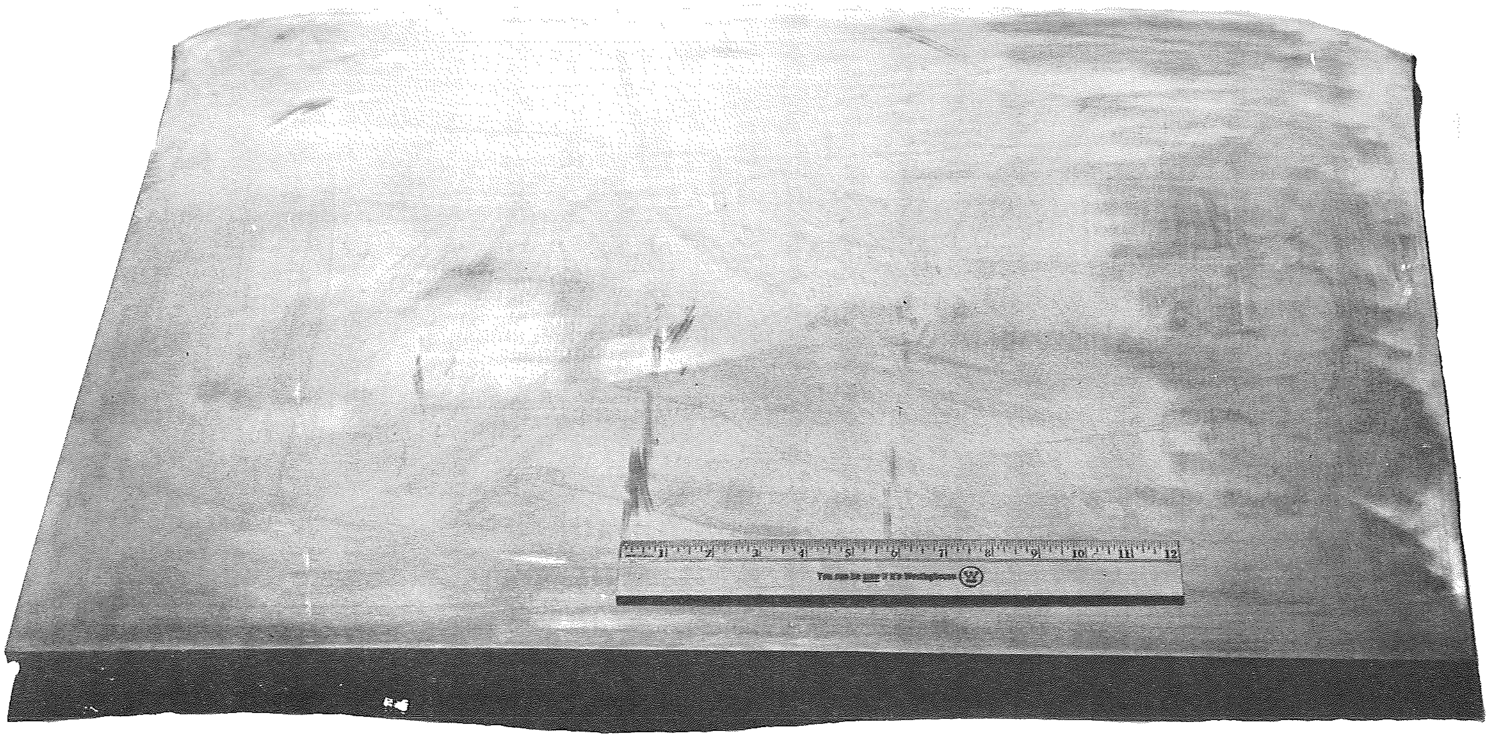
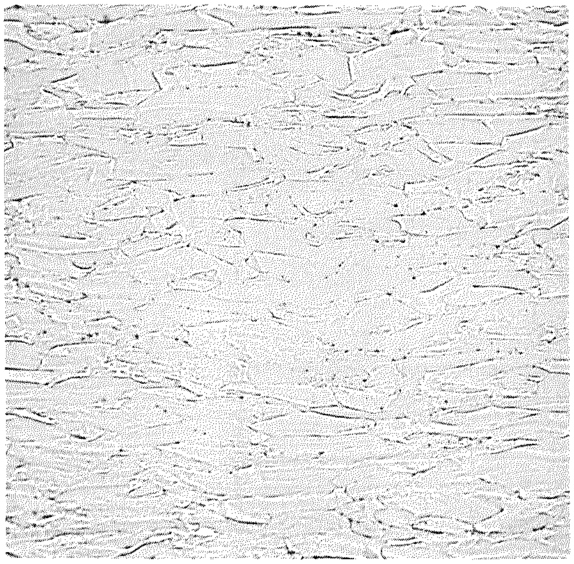
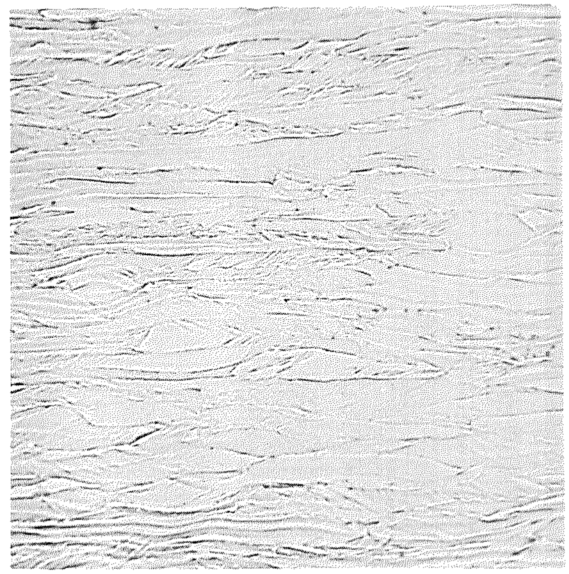


Figure 4-12. Rolled and Annealed T-111 Plate

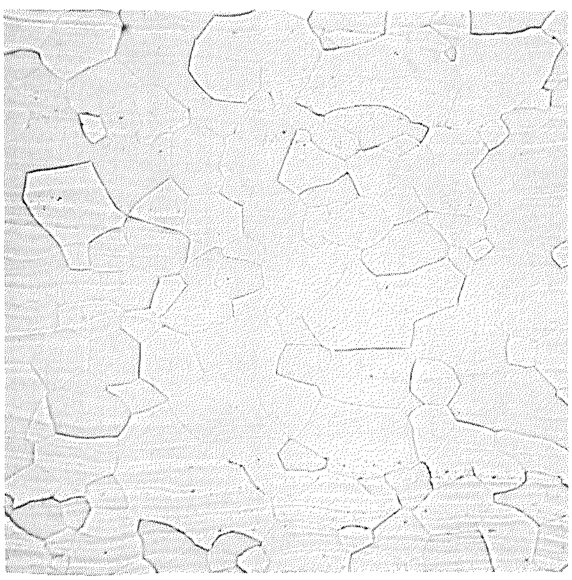


19,312 transverse 200X



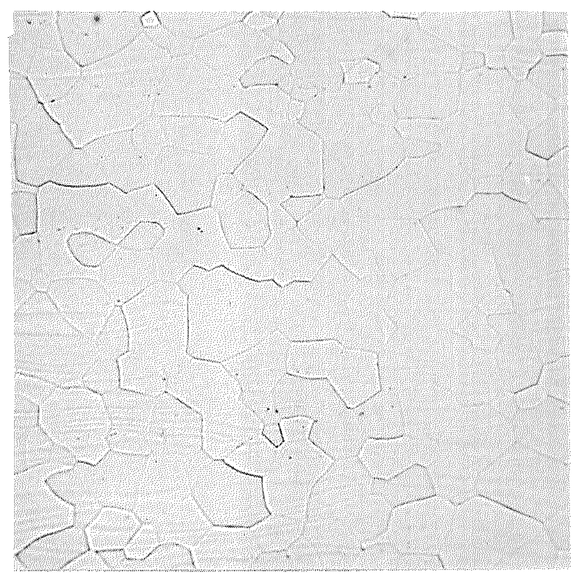
19,311 longitudinal 200X

As Rolled 65%



19,314 transverse 200X

A.S.T.M.
G.S.
7-8



19,313 longitudinal 200X

Annealed 1 Hr. 3000°F
210 DPH (30 Kg)

Figure 4-13. Comparison of Longitudinal and Transverse Sections from T-111 Plate

Table 4-4. Interstitial Level of T-111 at Various Stages of Processing

Sample Location	(interstitial level ppm)			
	C	O	N	H
Extrusion	25	30	11	---
Annealed Extrusion	35	45	7	---
Annealed Sheet Bar	48	25	29	0.7
Rolled Plate	48	51	20	1.2
Annealed Plate	40	40	40	1.0

After annealing, the rolled plates were inspected by ultrasonic and liquid dye penetrant techniques and found to be defect free. The surface finish of the as-rolled and annealed plates was better than 16 rms. Each plate was machined to 16.175 inches in width and was approximately 30 inches long by 0.425 inches thick. The 16.175 inch width allowed press forming a 5 1/2 inch diameter cylinder and provided 0.1 inch of stock for the machining of the mating edges for proper closure fit.

Press forming of the annealed T-111 plate was done at the Swepco Tube Corp.* on a 1500 ton capacity mechanical press equipped with a 20 foot long die. A 150 ton capacity six inch closed die press was used to complete the final sizing operation. The sequence used for forming the T-111 tube shells is illustrated in Figure 4-14 and the formed T-111 tube shells are shown in Figure 4-15.

Prior to forming the 30 inch lengths of T-111 plate, 5 1/2 inch diameter tube shells were first press formed from 3/8 inch thick x 24 inch long type 304 stainless and then a 5 inch length x 0.4 inch thick T-111 to demonstrate the suitability of the forming operation.

The very smooth (< 16 rms) surface of the T-111 plate prevented an adequate die grip during the press forming operation. Thus a larger than optimum bite was required on the initial forming operation resulting in a series of small radii around the tube circumference rather than a continuous circle. This necessitated additional work in the closed die forming operating to smooth out these irregularities. The clearance between mating longitudinal edges was 1.6 inches and provided access to the final edge machining operation. Maximum outer fiber strain of the formed tube shells was claculated to be 4.2%, although local areas may have been worked more severely during the final closed die sizing operation.

The formed tube shells were free of surface defects as indicated by dye penetrant inspection. Following the forming operation, the tube shells were pickled in 60 w/o water, 30 w/o HNO_3

*See Appendix 1.

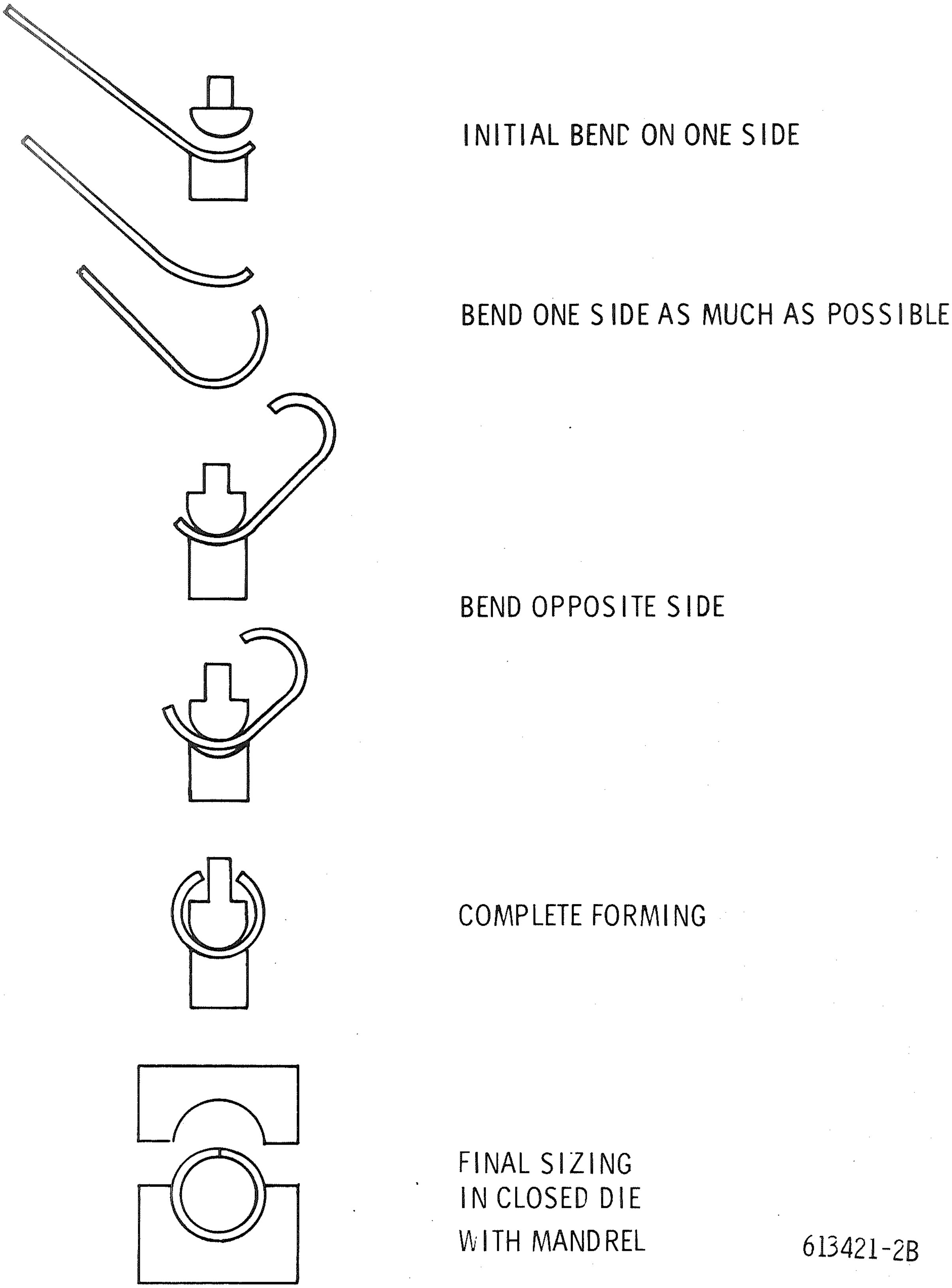


Figure 4-14. Tube Shell Forming Operation

Figure 4-15. Press Formed T-111 Tube Shells P, B, and C

and 10 w/o HF, and wrapped in 2 layers of tantalum foil in preparation for annealing. The tube shells were annealed for 1 hour at 3000°F to provide a stress free structure for welding.

4.5 Tube Shell Welding

The press formed and annealed tube shells were longitudinally seam welded using single pass electron beam welding without filler metal addition. The electron beam welding was done using a 30 KW Sciaky electron beam welder located at the Mech-Tronics Corp.* A typical as-welded tube shell is shown in Figure 4-16, and the welding parameters are summarized in Table 4-5.

A minimum gap clearance, necessary for electron beam welding, was achieved by precise final edge machining and careful alignment in the welding fixture. After machining the tube shells were placed in the welding fixture and the mating edges were clamped together (See Figure 4-17).

One inch diameter screws torqued to 200 ft-lbs provided a side clamping force of over 20,000 lbs. A 60,000 lb. capacity tensile test machine was used to augment the screw driven side clamps. The T-111 tubes were heated to 400°F during the alignment and clamping operation.

To ensure a full wall thickness weldment, 0.062 inch thick T-111 strips were clamped to the inside and outside diameter of the formed tube shells (See Figure 4-18).

Final alignment of the joint with the electron beam was done with the top filler strip removed. After alignment was completed, the top strip was replaced and the weld made. The bottom filler strip and a 3/8 inch thick T-111 backup bar were held in place by internal screw jacks.

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*See Appendix 1.

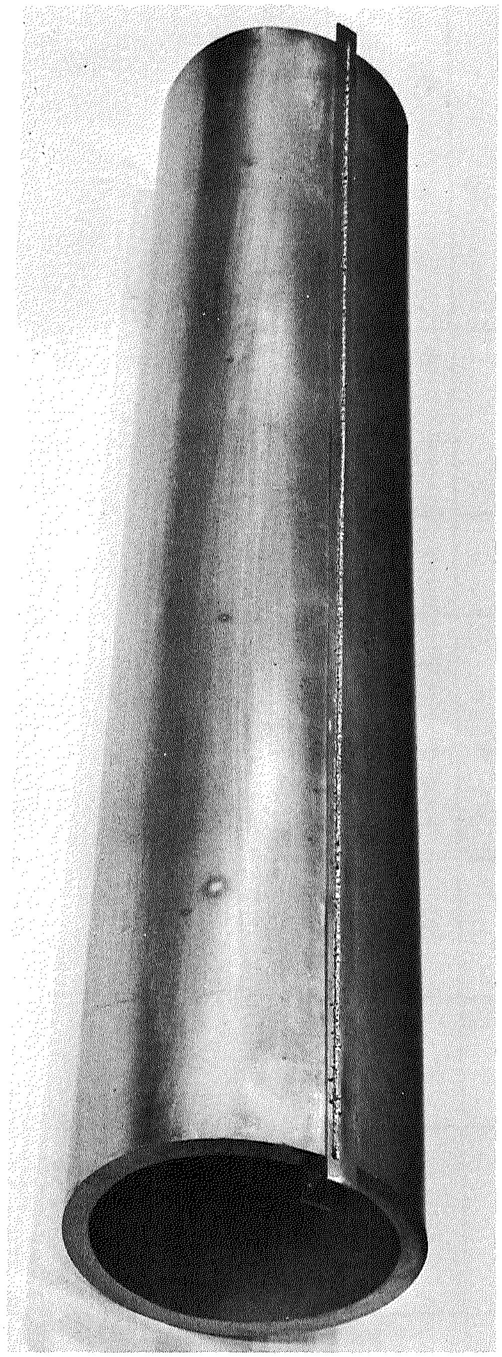


Figure 4-16. Electron Beam Welded T-111 Tube Shell B

Table 4-5. Conditions of Electron Beam Welding for Press Formed 5 1/2" Diameter T-111 Alloy Tube Shells[a]

Length of Cylinder Welded inches	Total Thickness of Seam inches	Voltage KV	milliamps	Power KW	Power Input KJ/inch	Focus[c]	Ratio of Beam Travel inches/min	
5	0.525	27	265	7.15	42.9	817	10	Insufficient penetration at start Top bead irregular with deep undercutting
30 (Cylinder C)	0.544	27	283	7.64	45.8	817	5+5[b]	Excessive penetration through T-111 backup bar
29 (Cylinder B)	0.544	27	240	6.49	38.9	827	5+5[b]	Penetration and top bead appearance acceptable

[a]All welding done at $\leq 5 \times 10^{-5}$ torr

[b]Electron beam travel of 5" per minute plus table travel of 5" per minute.

[c]Focus coil current setting as given in dial reading.

Note: Welding done at Mech-Tronics Corp., Chicago, Illinois, Division of Fansteel Metallurgical Corp.

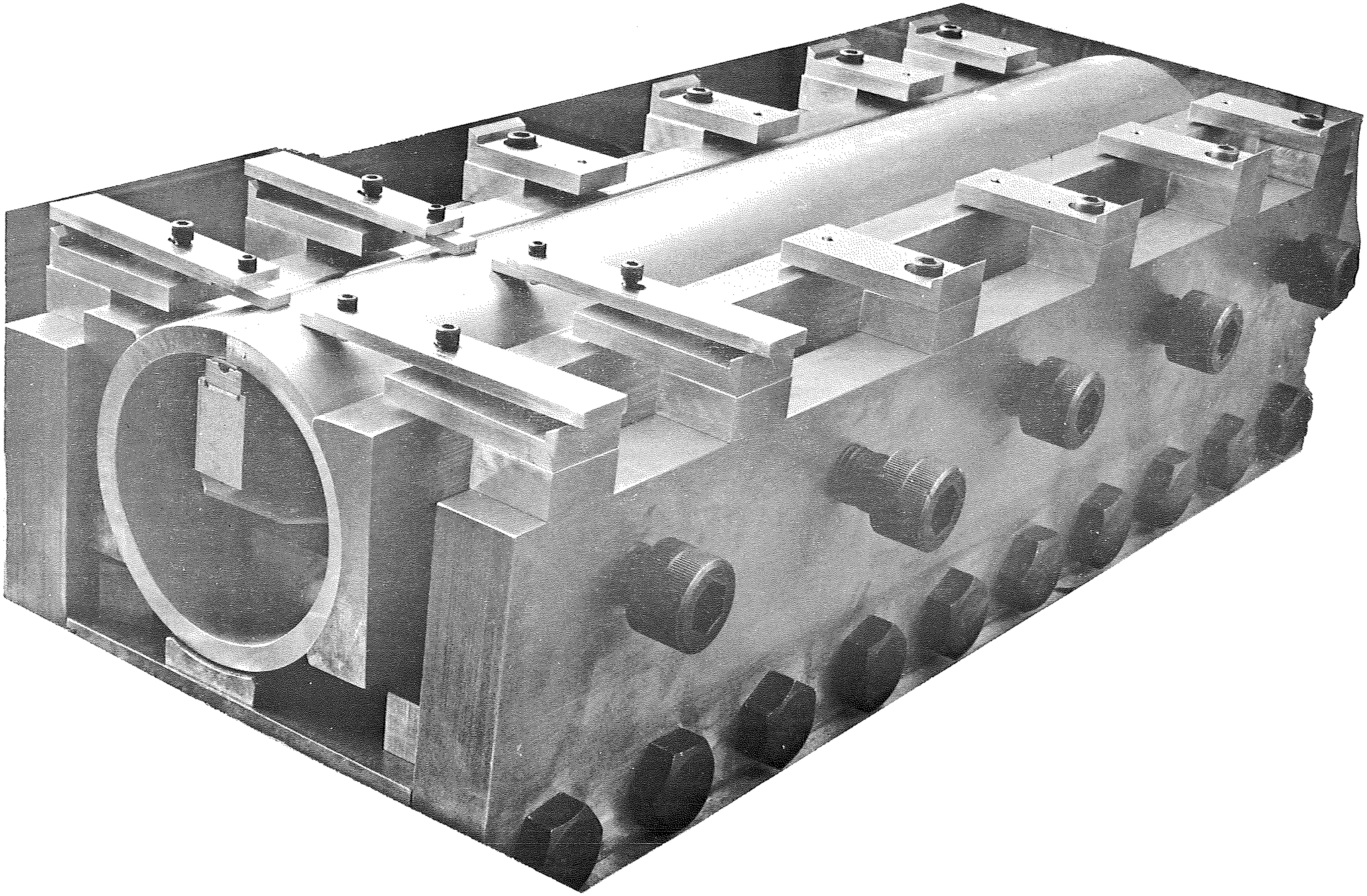


Figure 4-17. Tube Shell Weld Clamping Fixture

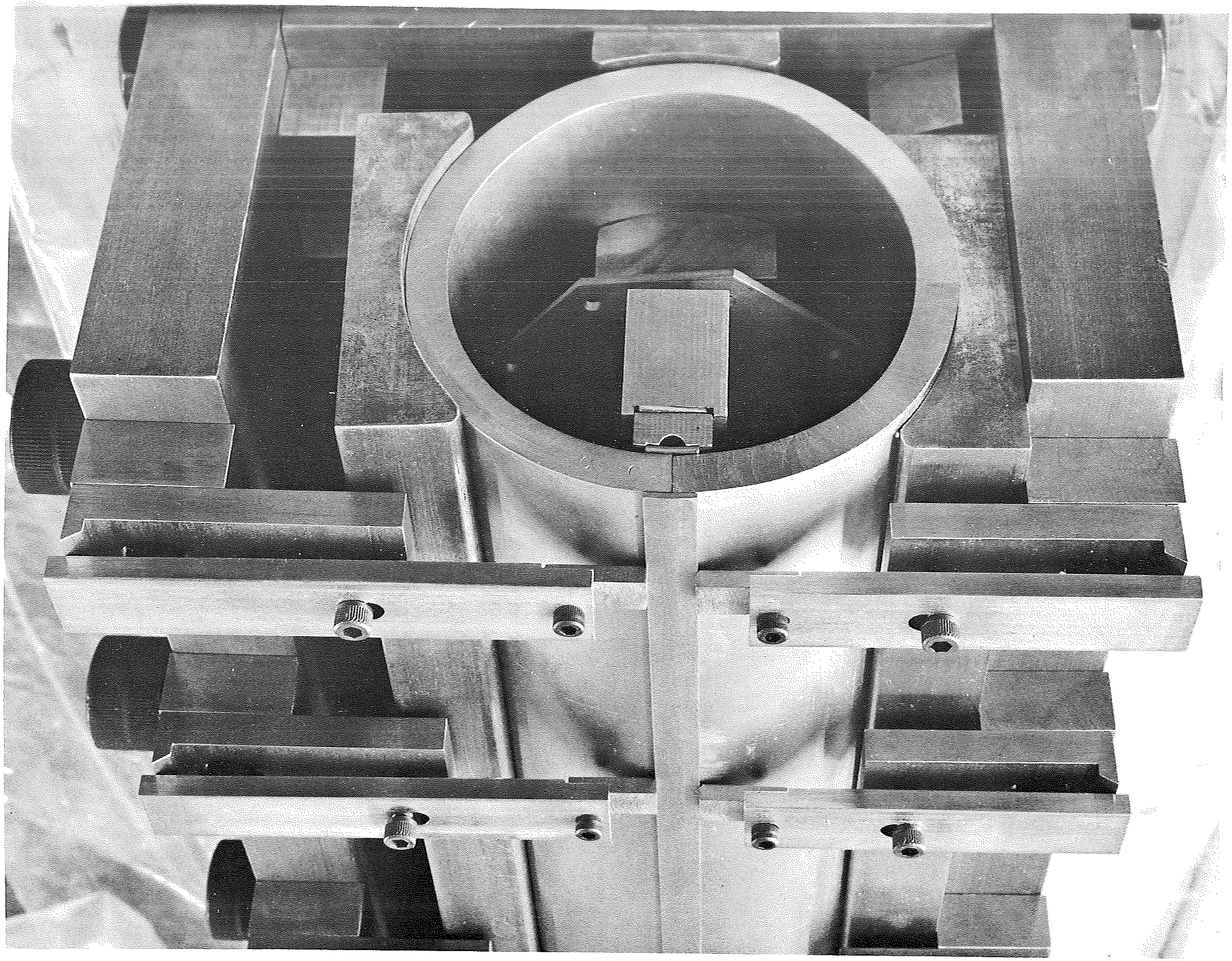


Figure 4-18. T-111 Tube Shell with OD and ID Filler Strips in Place

The final electron beam welding parameters were selected to provide as wide a joint as practical to accommodate any seam misalignments. The 10 inches per minute welding speed at 30 to 44 k joules (corresponding to 180-260 ma beam current at 27 kv) input per inch of weld were selected after preliminary evaluation of over 50 practice welds. Multipass GTA welding with filler metal addition was also evaluated, but a weld underbead microcracking problem was uncovered which eliminated this process from consideration. Details of the GTA multipass welding of thick section T-111 are described in Appendix 3.

Prior to welding of the two full length tube shells ("B" and "C") the 5 inch length tube (identified as "P") was welded to confirm the weld parameter selection (See Table 4-5). Marginal penetration indicated that slightly more power would be required. Also heavier T-111 filler strips were being utilized (0.062 inch vs 0.050 inch) on tubes "C" and "B". The parameters listed in Table 4-5 were selected for tube "C" and from the data plotted in Figure 4-19 appeared reasonable. However, excessive penetration occurred and the electron beam penetrated the T-111 backup bar and cut into the mild steel fixture frame supporting the backup bar. Chemical analysis (XRF, microprobe and spark source mass spectrometer) results indicated iron contamination was confined to the 1 - 2 inch length at the start and weld finish which were removed. That this was indeed the case was verified during the subsequent processing and property evaluation.

Prior to the welding of tube shell B, additional trial welds were made at significantly lower power settings and the final weld was made at 27 KV and 240 ma. In addition, a 0.440 inch thick bar of Ta-10W was placed between the T-111 backup bar and the steel fixture frame. The electron beam, however, did not penetrate the T-111 backup bar during welding of tube shell B.

No obvious reason was ascertained for the change in power requirements between the many preliminary welds and the welding of tube shells C and B. The electron gun was dismantled, cleaned, and realigned in the 2 months between the preliminary welding and the final welds.

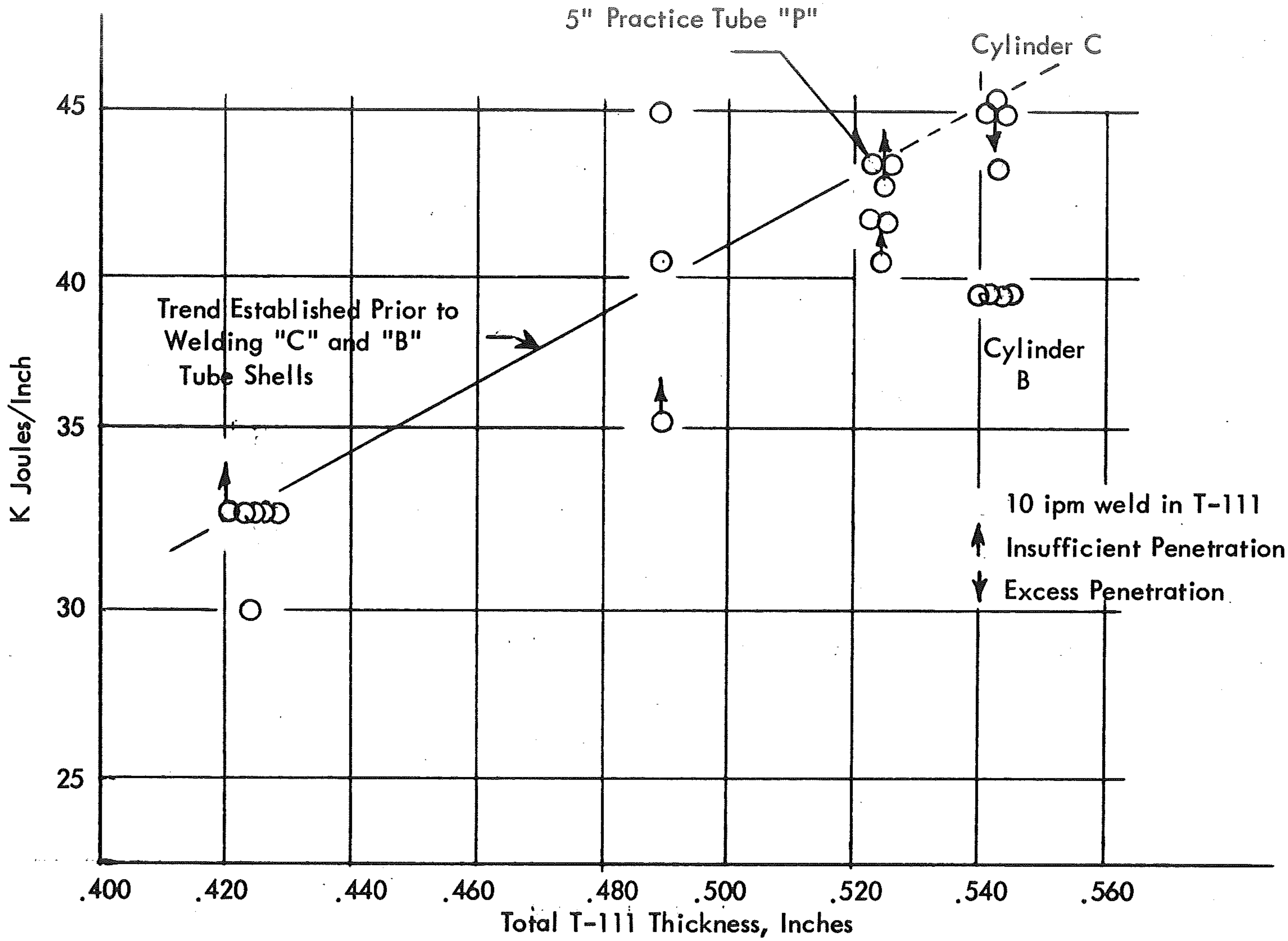


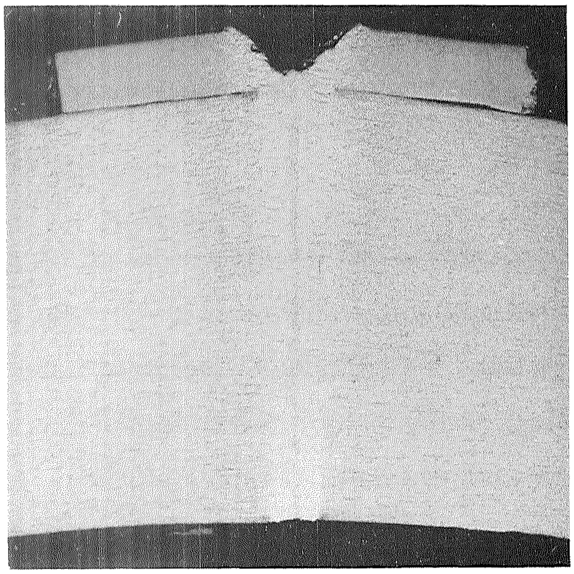
Figure 4-19. Power Input Per Inch Vs. Thickness of T-111 Plate at 10 ipm Welding Speed

Subtle changes in the gun characteristics resulting from this operation may have caused an increase in beam power density.

The weld filler strips were removed from the OD by end milling and from the ID by grinding. After weld conditioning, the tube shells were inspected by liquid penetrant and radiographic techniques. The welds were free of surface indications and the radiographs indicated sound welds along the entire length except for a single defect (0.010 inches) .3 inch from the weld start end of tube shell "C" and one 3/4 inch from the weld finish end of tube shell "B". These defects were removed during preparation of the tube shell ends for extrusion attachments needed for the tube reduction operation. A reduction in diameter of from 0.025 to 0.045 inches was observed following welding.

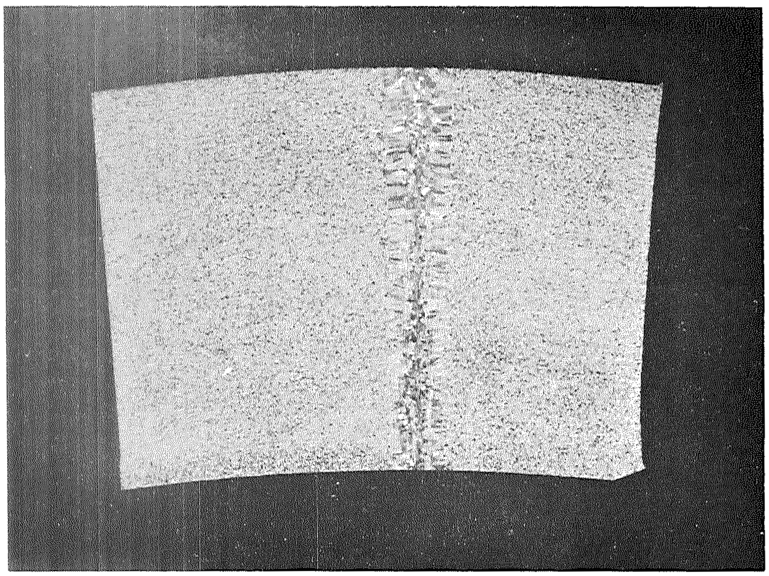
The welded tubes were then cleaned, pickled, rinsed, wrapped in clean tantalum foil and annealed for one hour at 3000°F at $\leq 5 \times 10^{-5}$ torr.

Transverse macrosections of the as-welded and annealed tube sections are shown in Figure 4-20. The program goal for the completed weld reduced and heat treated tubing was to obtain a completely homogenized weld structure of fine grain size indistinguishable from the base metal. An experiment was conducted to develop the post weld and interpass annealing schedule to produce a homogenized, fine grained weld structure. Typical reduction schedules and annealing treatments required to produce 4 1/4 inch and 3 inch diameter tubing were evaluated. With consideration to annealing furnace capability, the best combination was a 3000°F post weld anneal followed by 3000°F interpass anneals. Selection of the thermo-mechanical processing will be discussed in a later section.



As Welded Bottom Filler Strip Removed

20,206 5X



Annealed 1 hr. 3000°F

20,577 5X

Figure 4-20. Transverse Wall Sections of Tube Shell C Comparing the Welded and Annealed Weld Structure

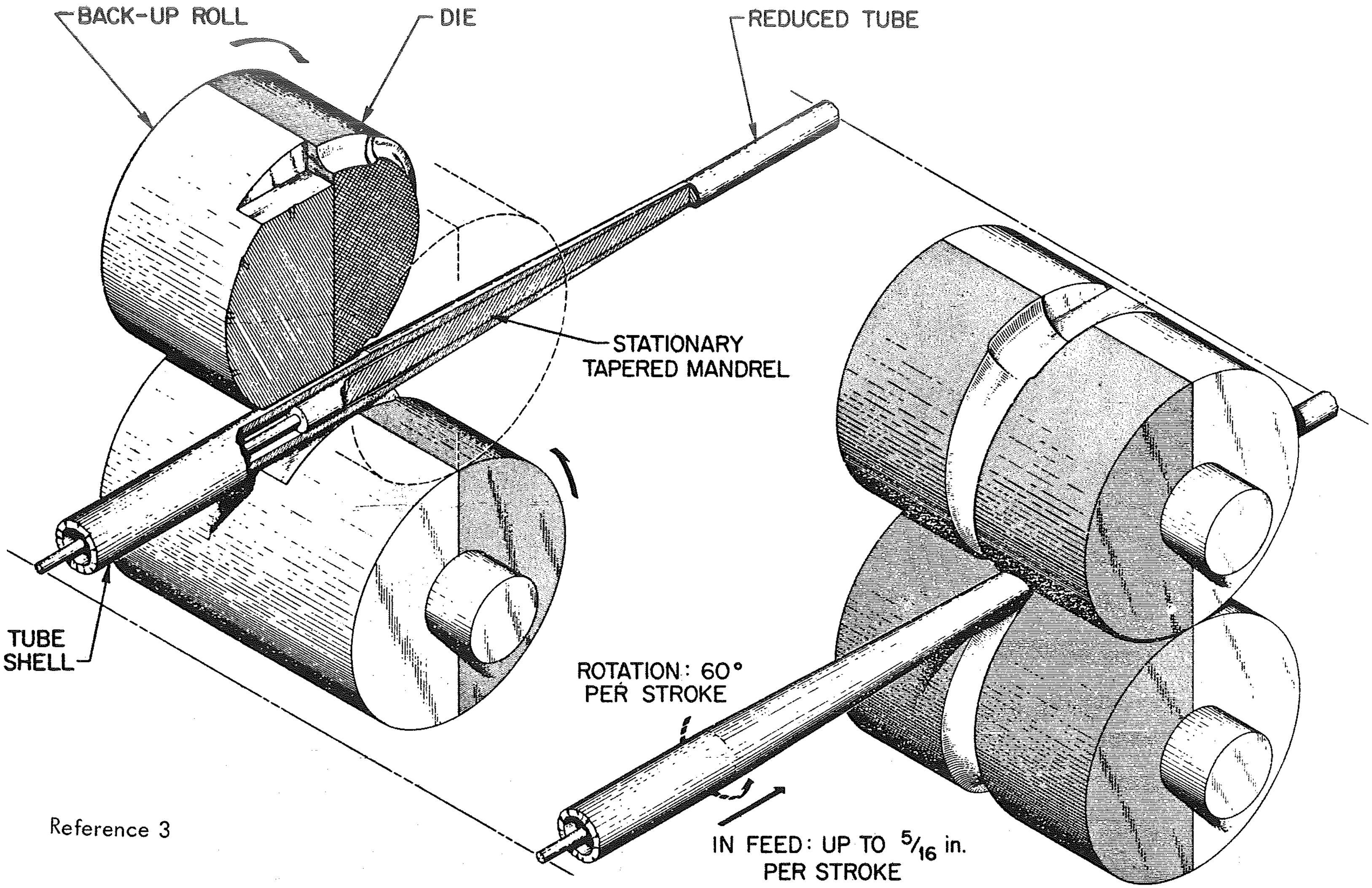
5. TUBE REDUCTION

The seamless and welded tube shells (5 1/2 inch diameter x 1/2 inch thick wall) were tube reduced in 2 passes to 4 1/4 inch OD x 1/8 inch wall tubing and in 4 passes to 3 inch OD x 0.080 inch wall tubing. All tube reduction was done at room temperature on a Roto roll or cold pilger type machine (See Figure 5-1) located at the Wooster, Ohio plant of the Timken Roller Bearing Co.* Tube reductions were accomplished on a double roll, rocking die tube reducer using a tapered stationery mandrel as internal support. This feature permits simultaneous reduction of the diameter and wall thickness with total reductions of up to 80% per pass. Approximately a 2 foot length of tube is in the working area of the die at one time and the tube is rotated 30° and advanced 1/8 inch in each stroke. All reductions were done at room temperature with a flowing water soluble lubricant on the outside surface and a soap lubricant on the inside, or mandrel surface. This lubrication practice is used for producing alloy and stainless steel tubular products.

5.1 T-111 Tube Reduction Characteristics

Both the seamless and welded tube shells were satisfactorily reduced to 4 1/4 and 3 inch diameter tubing following the reduction sequences shown in Figures 5-2, 5-3, and 5-4. The final as-reduced tubing is shown in Figures 5-5 and 5-6. Prior to each reduction stage, small test lengths of tubing were worked to provide an on the spot evaluation before committing the program material. Excellent workability was exhibited by the seamless and welded tube shells. There were no material losses from either the seamless or welded tube shells during processing which could be attributable to the tube reducing process. Between each reduction pass, the tubes were cleaned, pickled, rinsed, wrapped with tantalum foil, and annealed for 1 hour at 3000°F at $< 5 \times 10^{-5}$ torr. Single reductions as high as 65% were accomplished with no apparent difficulties. Dimensions of the final tubing were well within the commercial tolerances for this size range as illustrated by the data in Table 5-1.

*See Appendix 1.



Reference 3

613463-10B

Figure 5-1. Tube Reducing Process

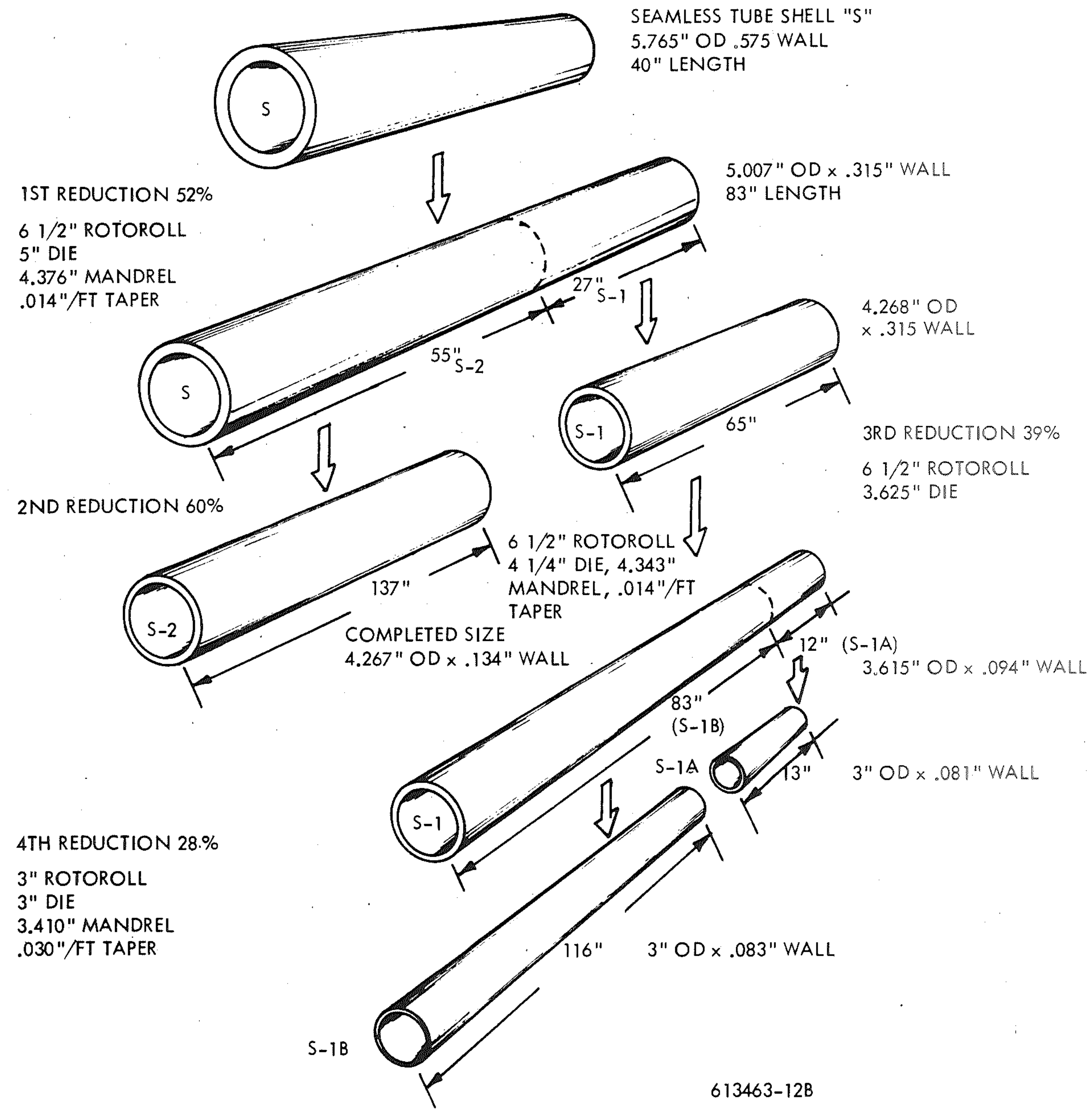


Figure 5-2. Processing Sequence for Seamless Tube Hollow to 4 1/4" OD and 3" OD Tubing

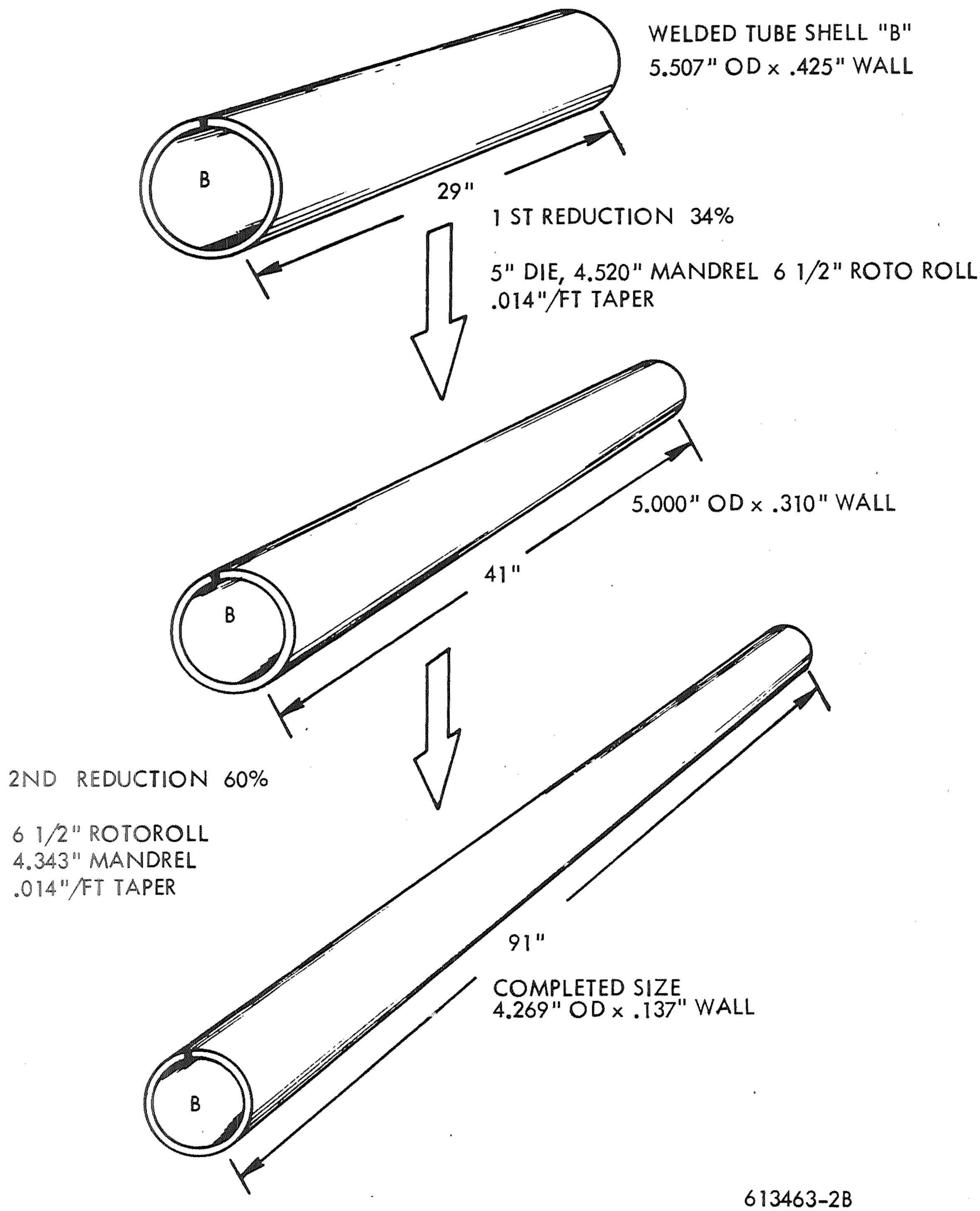


Figure 5-3. Processing Sequence for Welded Tube Hollow "B" to 4 1/4" OD Tubing

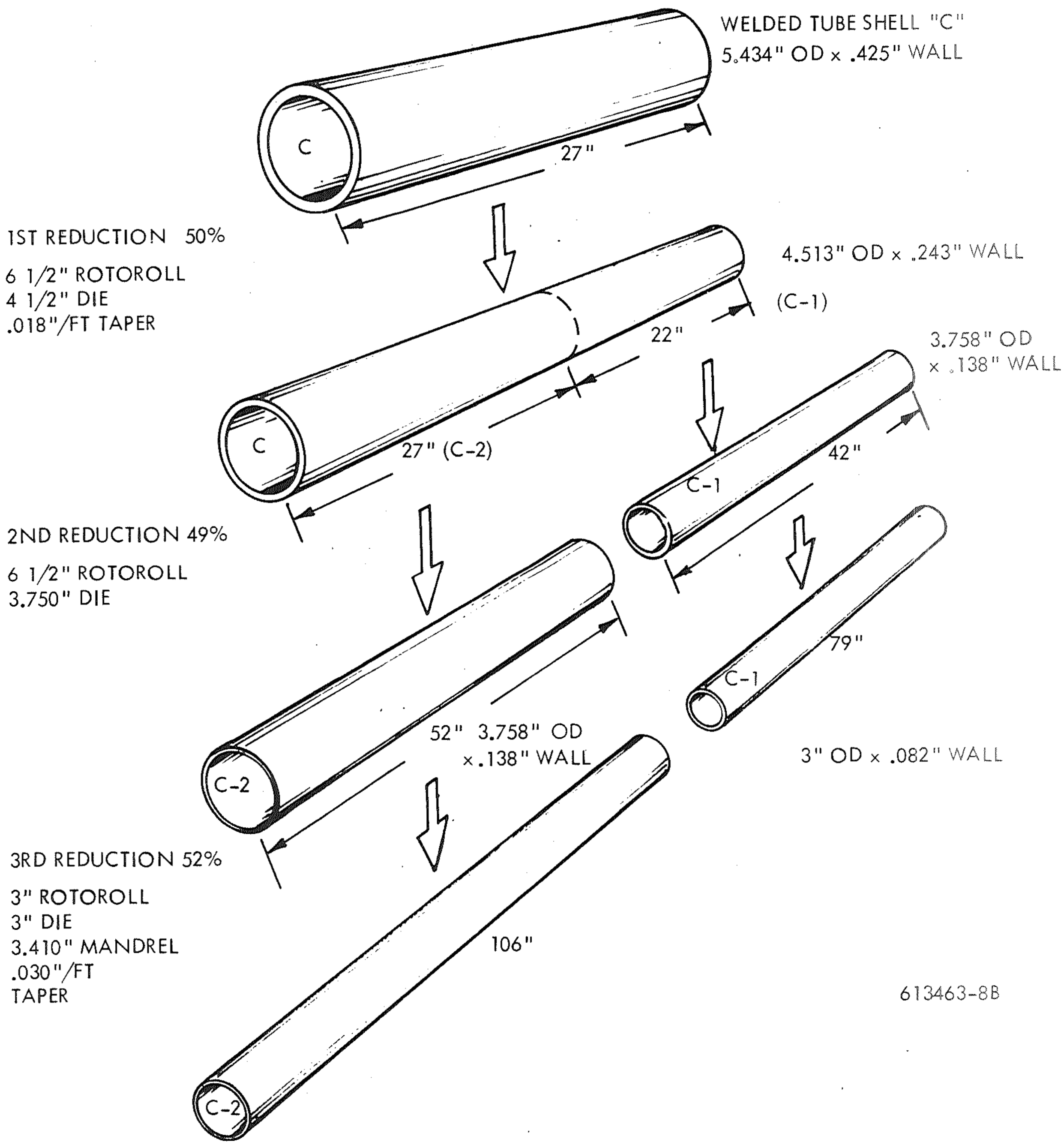


Figure 5-4. Processing Sequence for Welded Tube Hollow "C" to 3" OD Tubing

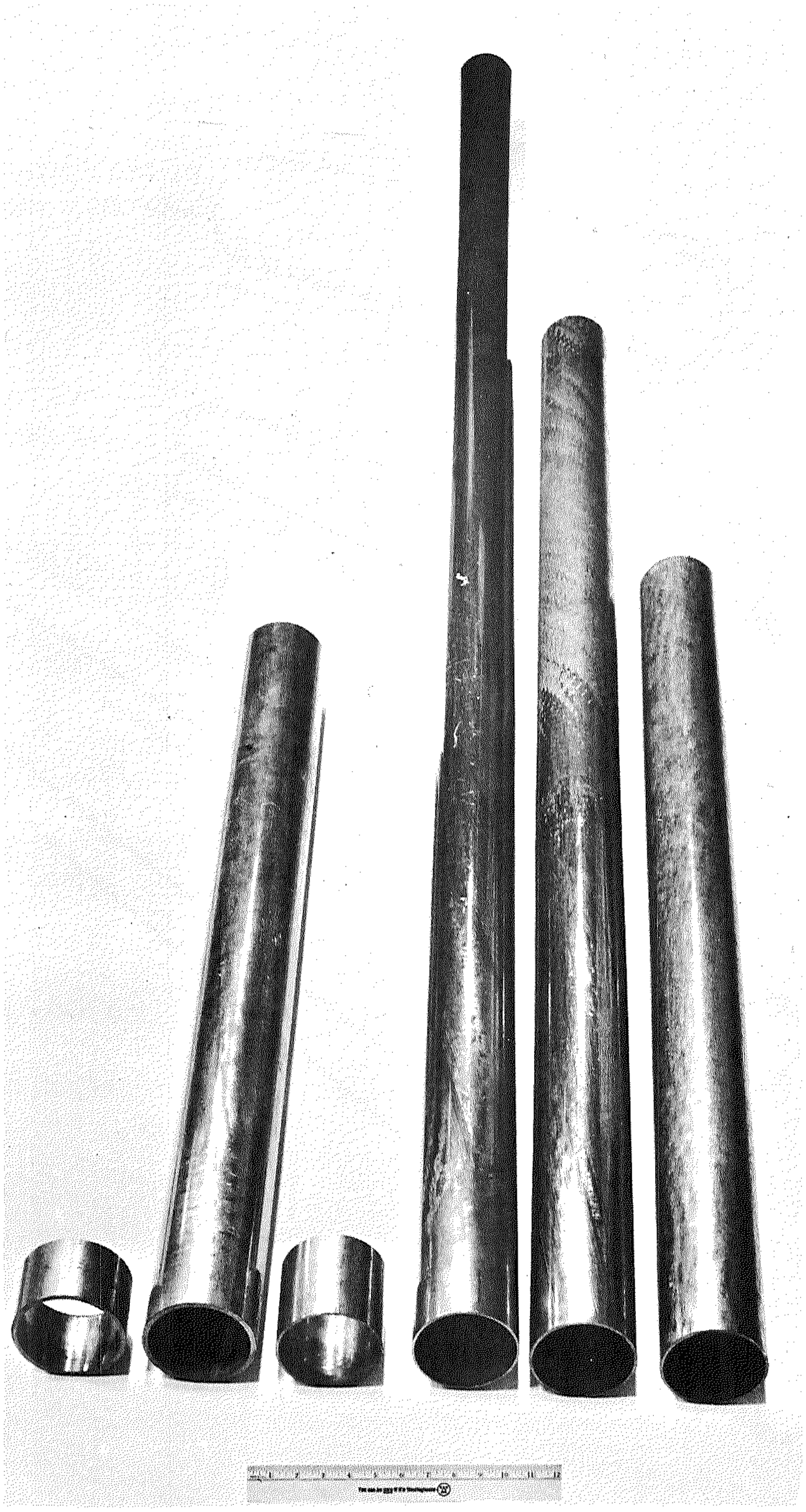


Figure 5-5. Tubes T, C, P, S-2, and S-1 As Reduced to 4 1/2" and 4 1/4" OD

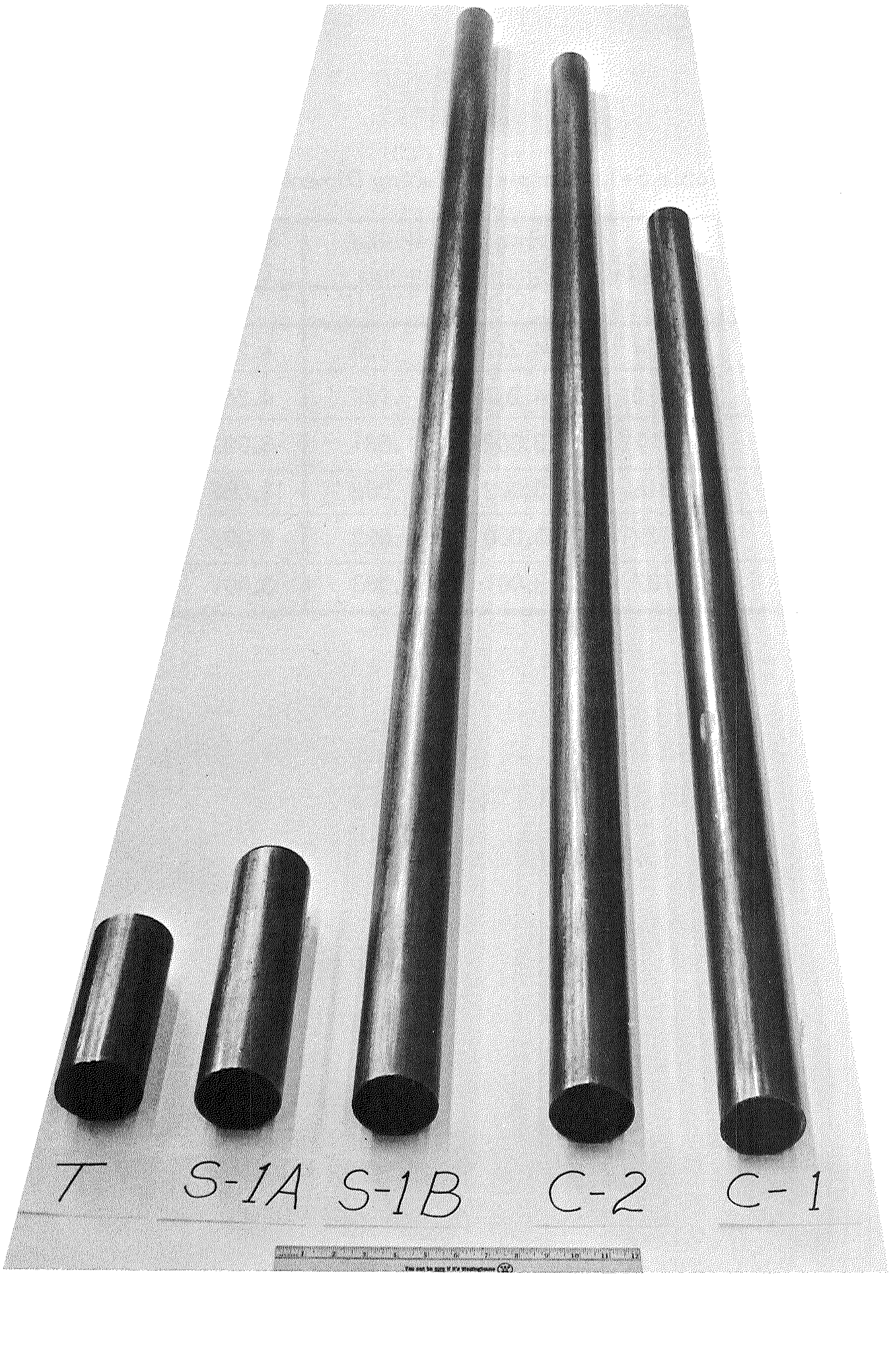


Figure 5-6. Tubes T, S-1A, S-1B, C-2, and C-1 As Reduced to 3" OD x .080" Wall

Table 5-1. Completed Tubing Dimensions

Tube No.	Length inches	Front, Numbered End Dim. Inches		Rear, Opposite End Dim. Inches	
		OD	Wall	OD	Wall
S-2	134	4.267	.138	4.248	.122
B	90	4.268	.139	4.268	.138
S-1A	12	3.002	.081	3.002	.081
S-1B	116	3.002	.082	3.002	.082
C-1	79	3.000	.092	3.000	.082
C-2	107	3.001	.083	3.001	.083

The limited scope of this investigation and the utilization of existing technology did not permit optimization of either lubrication practice or the reduction schedule. As noted earlier, the T-111 tube reductions were accomplished on cold pilger equipment designed and used for the high volume production of low alloy and stainless steel tubular products. The lubrication practices employed for the T-111 processing were those developed and used for low alloy and stainless steels and consisted of Armour beef tallow soap on the OD and ID. Each of the tube reducing machines are incorporated into a factory wide, closed loop water base soluble oil coolant system. Thus Timken would not deviate from their standard lubrication practice to avoid affecting the scheduled tubing production. Several lubricants were compared with the Armour beef tallow soap using a ball-on-plate simulated wear test. However, none of the lubricants showed any advantage over the lubricant being used.

5.2 Lubricant Performance

A high quality as-tube reduced outside diameter surface finish was obtained on only one tube (Tube S-2). The remaining tubes were completed with surfaces varying from slightly rough to rough and fretted although the same lubrication technique was used for each tube except the final reduction to 3 inch diameter. A high pressure gear lubricant was added at this stage with mixed results. No correlation was observed between the several reduction variables and surface finish and as shown in Table 5-2, which details the surface finish and lubrication techniques used. The mate to tube S-2, tube S-1, was processed identically, but developed a rough surface finish. Variables such as die wear may be a factor since tube S-1 was processed immediately following S-2.

A conversion coating (a standard surface treatment for stainless steels and titanium[5]) would lessen galling tendencies by preventing clean metal-to-die contact. Preliminary attempts to apply a conversion coating to T-111 were unsuccessful and were not able to be explored in any detail within the scope of the program. However, anodizing, either electrolytically or by air oxidation at 800°F may be beneficial and is an area which should be investigated in future work. It should be noted that the severe galling encountered during this investigation was not observed during processing of small diameter (2 inch diameter) tube blanks[2].

Table 5-2. Surface Appearance and Lubrication Practice for Tube Reduction Sequences

	First Reduction		Second Reduction		Third Reduction		Fourth Reduction	
Tube	Surface	Lub.	Surface	Lub.	Surface	Lub.	Surface	Lub.
S	Rough 75 RMS	A						
S-2			Excellent smooth < 32 RMS	A				
S-1			Rough fretted < 125 RMS	A	First 1/4 smooth 32 RMS	A		
S-1A							Rough 125 RMS	A+B
S-1B							Rough 125 RMS	A+B
B	First 2/3 smooth < 50 RMS Last 1/3 rough fretted > 190 RMS	A	First 1/3 smooth 32 RMS Last 2/3 rough 125 RMS	A[1]				
C			Surface good at start and finish	A				
C-1			< 32 RMS, bad in center > 125 RMS		Rough > 125 RMS	A	Rough 125 RMS	A+B
C-2					Rough > 125 RMS	A	First 1/2 rough 125 RMS Last 1/2 32 RMS	A+B
P	Smooth < 50 RMS	A	Smooth < 63 RMS	A				
T			Smooth < 63 RMS	A	Rough 125 RMS	A	Rough > 63 RMS	A+B

Prior to each reduction sequence the tubes were immersed in a heated solution of soap and detergent (proprietary) which produced a lubricating soap film on all surfaces.

A - Flowing water soluble oil on OD, Armour soap on mandrel.
B - Ironsides gear lube T GHHEP

[1] Coolant supply interrupted at transition to rough surface.

The optimization of the tube reduction schedule for T-111 tubing was not feasible during this program because of the limited quantity of material processed. Other constraints which did not permit reduction schedule optimization were of course the limitations imposed by the maximum ingot size and extrusion press capacity.

5.3 Discussion

From the results of this study, it appears that significant reduction in the tube inside diameter should occur concurrently with reductions in wall thickness. This would allow a taper on the mandrel of about 0.030 inch/inch which facilitates the tube to clear the mandrel after the working stroke. Mandrel sticking was experienced during the initial reductions where the mandrel taper was on the order of 0.014 inches/inch. Comparison of the work hardening behavior of T-111 with stainless steel (See Figure 5-7) indicates that single reductions as high as 80% may be possible providing proper lubrication is provided. The excellent workability demonstrated by T-111 during this investigation suggest that this is certainly feasible.

One other aspect worth discussing is that imposed by processing of relatively short pieces of material in large equipment. Normally, the minimum length piece going into the 6 1/2 inch capacity Roto-Roll machine is on the order of 10 feet. This length is necessary to bridge the gap between the work rolls and the take up collet. Since the longest starting T-111 tube shell was only 45 inches, the balance of the length was made up by the attachment of a mild steel follower as shown in Figure 5-8.

The initial tube reductions were made with pinned followers of the configuration shown in Figure 5-9a. The pins generally held until the joint area entered the reducing die at which point the tantalum lap joint fractured. On subsequent reductions, as the wall became thinner, pin joints were too weak and machine threads, designed to tighten with work rotation, were used. It is important to maintain a full wall thickness across the joint to prevent the wall from buckling or upsetting since the working stroke of the tube reducer applies considerable longitudinal pressure approaching that of the wall strength (See Figure 5-9b). Using minimum 10 foot length sections of course eliminates this problem entirely.

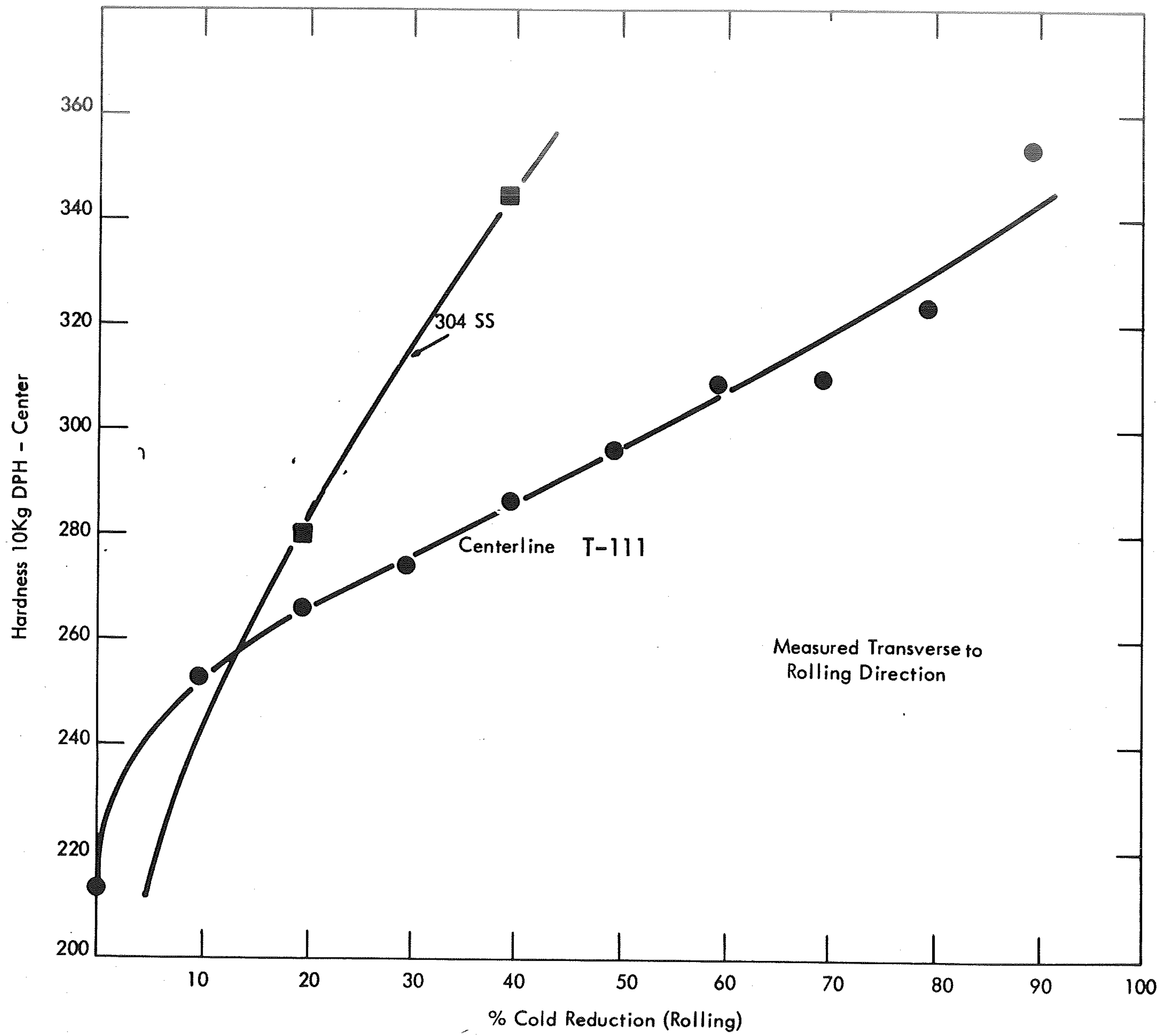


Figure 5.7. Hardness Versus % Cold Reduction for T-111 Plate

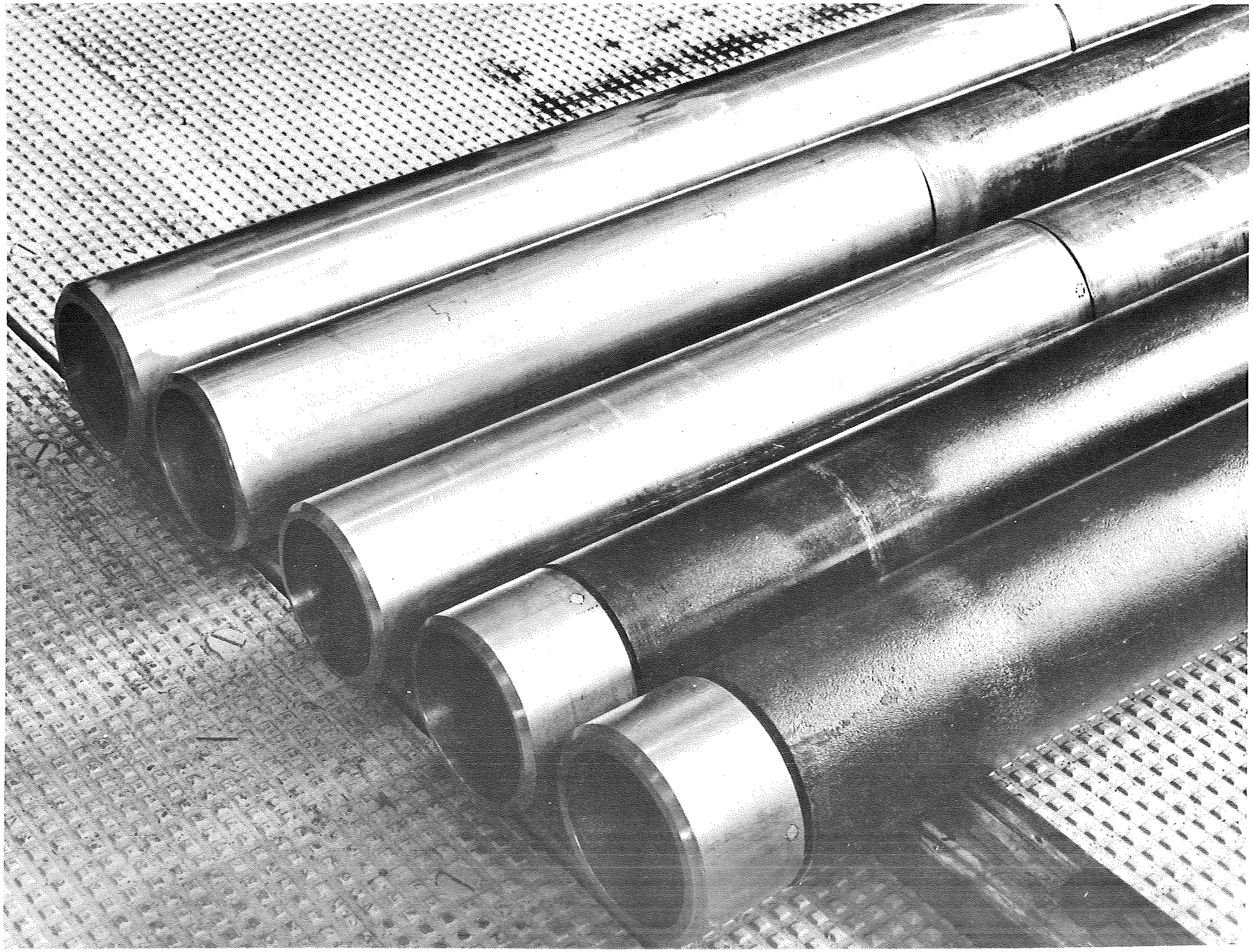


Figure 5-8. Tube Shells Prepared for Initial Reduction Sequence

a.

b.

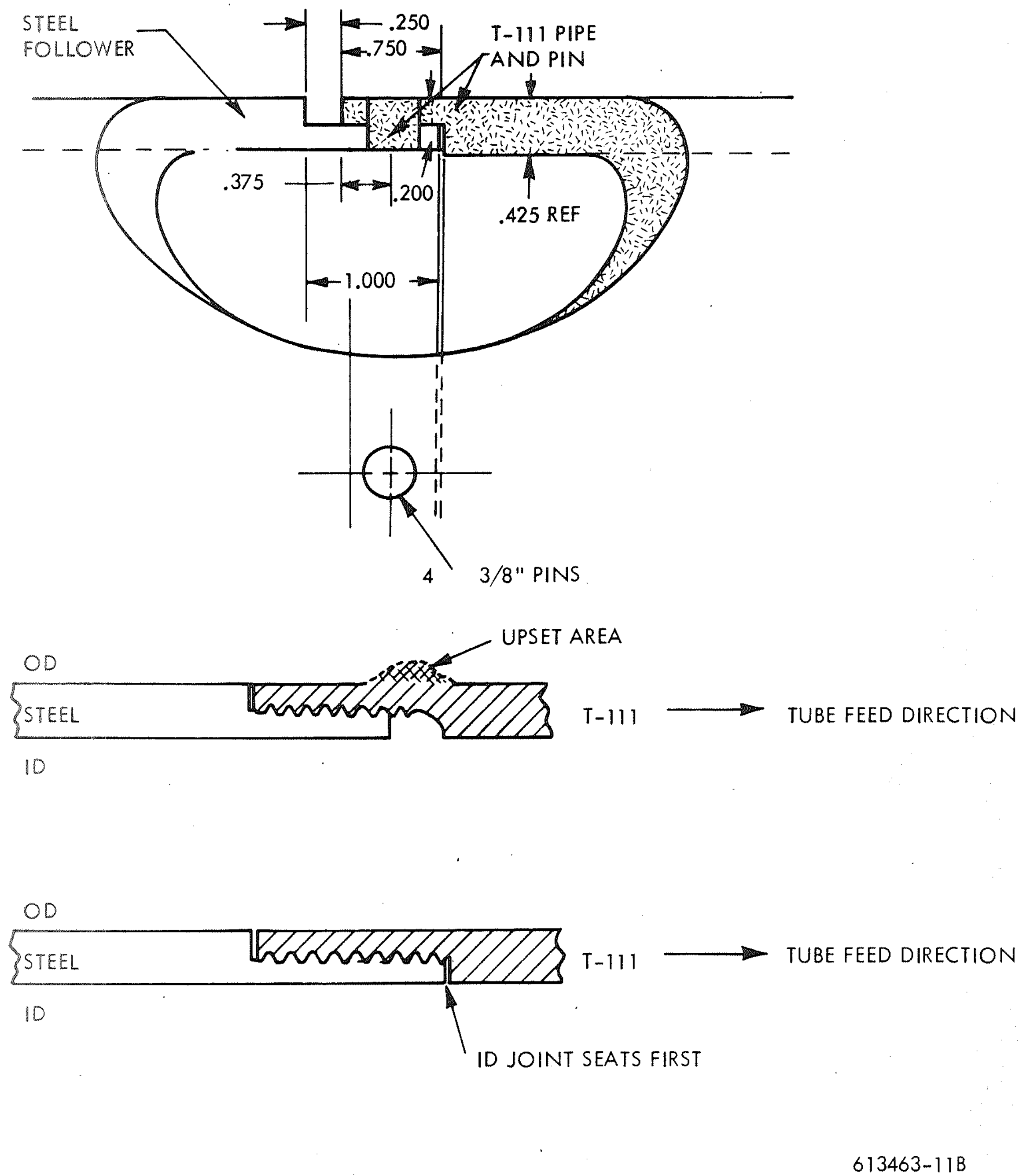


Figure 5-9. Attachment Techniques for Follower

6. TUBING EVALUATION

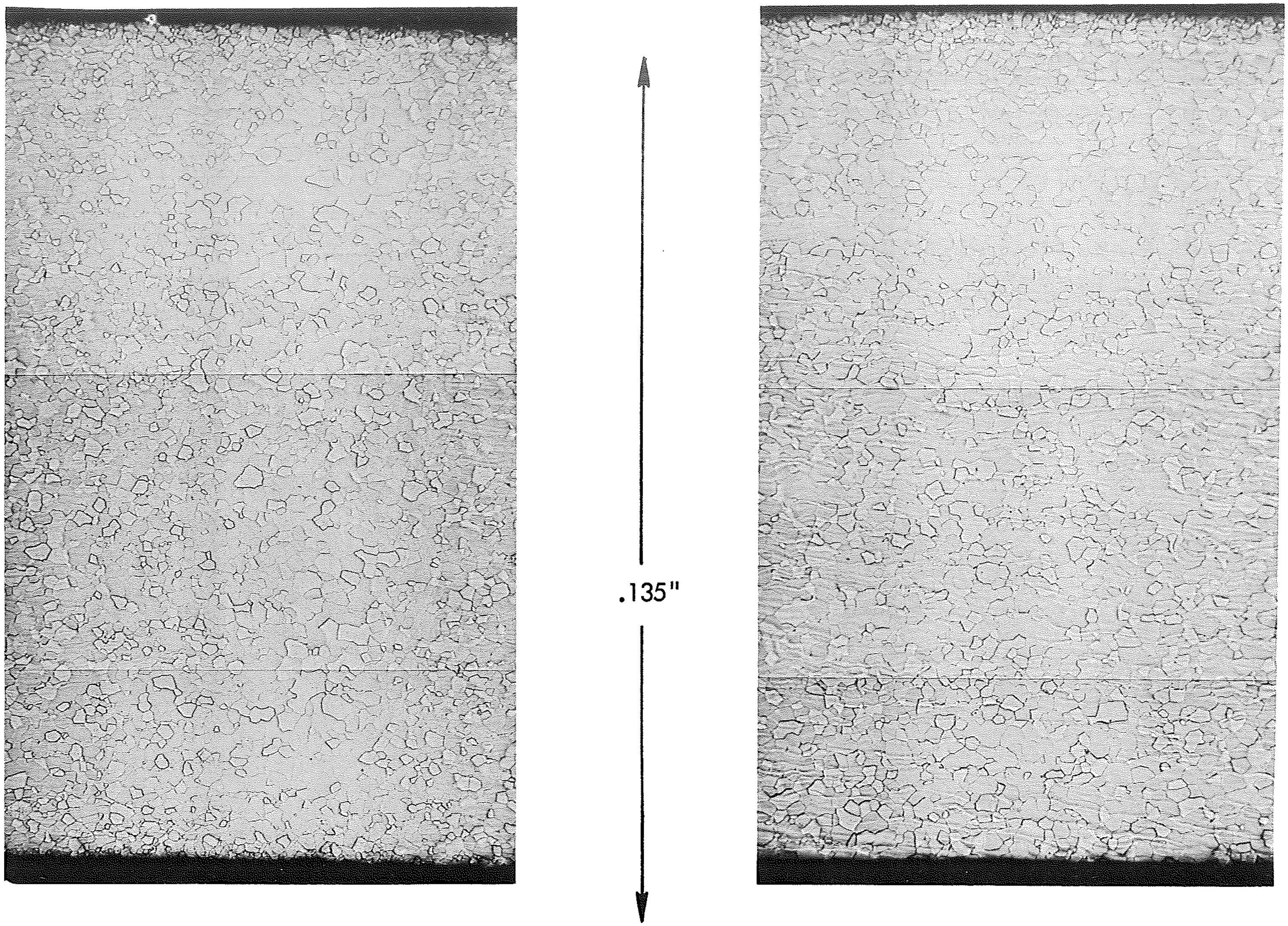
In addition to mechanical property evaluation, chemical analyses, and metallographic examination, the final tubing was liquid dye penetrant inspected and helium leak checked. The completed seamless and welded tubing was defect free as determined by helium leak testing and liquid penetrant inspection.

6.1 Microstructure

The microstructure of the seamless and weld-redrawn T-111 tubing after annealing 1 hour at 3000°F is shown in Figures 6-1 and 6-2. The full transverse tube wall sections shown here illustrate not only the uniformity of the recrystallized grain size but the effectiveness of the mechanical working and annealing sequence in eliminating evidence of the cast metal zone in the welded tube shells. The area of the weld in the final tubing can only be discerned by the absence of the "ghost pattern" which is evident in the adjacent base metal as well as in the seamless product. The "ghost pattern" or striated appearance of the etched sample is commonly observed in columbium and tantalum base alloys, although the exact cause as yet has not been satisfactorily explained.

Since T-111 does not undergo an allotropic transformation, grain size is controlled and refined by the combination of mechanical working and recrystallization annealing. As-worked T-111 recrystallizes to produce an equiaxed microstructure after annealing for 1 hour at 2500°F to 3000°F, depending on the amount and temperature of prior deformation[1,2] and the T-111 used for this study behaved rather predictably.

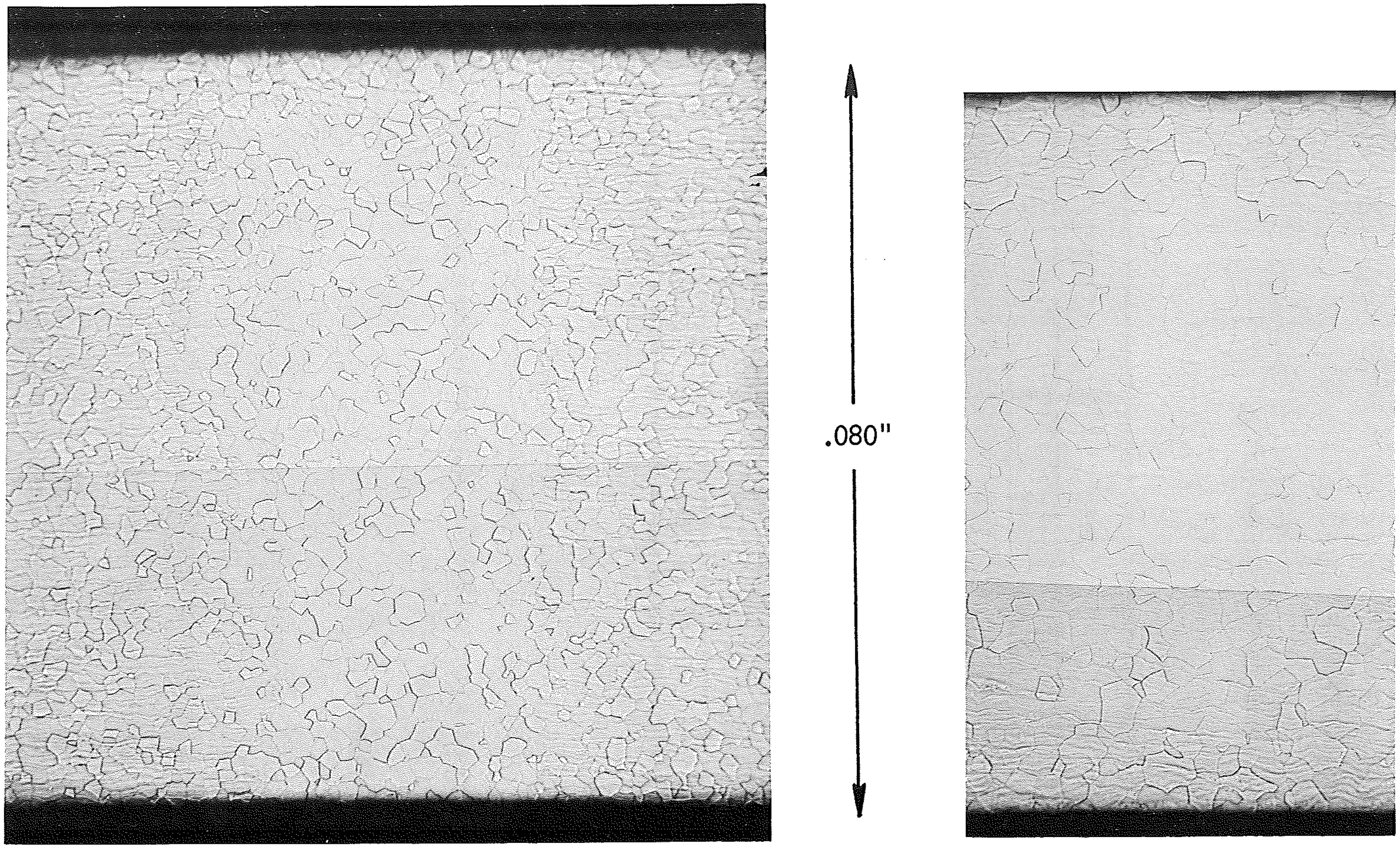
As shown by the grain size data in Tables 6-1 and 6-2, significant refinement of the as-cast grain structure was achieved by the various working and annealing operations. It should be noted that all working operations were done below the recrystallization temperature for T-111 and thus can be termed "cold work" even though the primary working operations were performed at 2000-2300°F. The hardness values of 200-210 DPH measured on the as-cast and recrystallized



20,885 ← Weld Zone → 40X 20,883 40X

Weld Reduced Seamless Tubing

Figure 6-1. Transverse Full Wall Sections of Annealed 4 1/4" OD Tubing Comparing Seamless And Weld Reduced Material



21,175 ← Weld Zone → 50X

WELD REDUCED TUBE C-1

21,165 50X

SEAMLESS TUBE S-1B

Figure 6-2. Transverse Full Wall Sections of Annealed 3" OD Tubing Comparing Seamless and Weld Reduced Material

Table 6-1. Grain Size and Hardness of Seamless Tubing

Processing Stage	DPH[1]	ASTM Grain Size[2]
Ingot for Seamless Tubing	208	-2 to -6
Extruded Tube Shell (As Extruded) (70% Red.)	266	not determined
Machined and Annealed Tube Shell	204	4 to 5
Tube S 5" OD x .300" Wall (52% Red.)	199	5
Tube S-2A 4 1/4" x .135" Wall (60% Red.)	202	6 to 7
Tube S-1 3 5/8" OD x .094" Wall		6
Tube S-1B 3" OD x .083" Wall (28% Red.)	199	4 to 5
Tube S-1A 3" OD x .083" Wall	200	5 to 6

[1] All hardness values as vacuum annealed 1 hour at 3000°F unless otherwise noted taken at load of 30 Kg on metallographically polished surface.

[2] Determined by the line intercept technique.

Table 6-2. Grain Size and Hardness of Weld-Reduced Tubing

Processing Stage	DPH[1] Base	DPH[1] Weld Metal	ASTM Grain Size[2] Base	ASTM Grain Size[2] Weld Metal
Ingot for Welded Tubing	213		-2 to -6	
5" Extrusion - As Extruded (70% Red.)	260		not determined	
5" Extrusion - Annealed	216		5 to 6	
Forging A As Forged (75% Red.)	279		not determined	
Forging A Annealed			7 to 8	
Rolled Plate As Rolled (66% Red.)	312		not determined	
Rolled Plate Annealed	210		7 to 8	
Tube C Welded and Annealed			6 to 7	4 to 5
Tube C 4 1/2" x 1/4" Wall - As Reduced (50% Red.)	273		not determined	
Tube C 4 1/2" OD x 1/4" Wall Annealed	209	215	6 to 7	5
Tube C 3 3/4" OD x .135" Wall Annealed (49% Red.)	212	214	6 to 7	5 to 6
Tube C 3" OD x .083" Wall Annealed (52% Red.)	208		6 to 7	6 to 7
Tube B Welded and Annealed			7 to 8	4 to 5
Tube B 5" x .300" Wall Annealed (34% Red.)			5 to 6	5 to 6
Tube B 4 1/4" x .135" Cold Worked (60% Red.)	294		not determined	
Tube B 4 1/4" x .135 Annealed	204	204	6 to 7	5 to 6

[1] Determined using 30 Kg load on metallographically prepared surface.

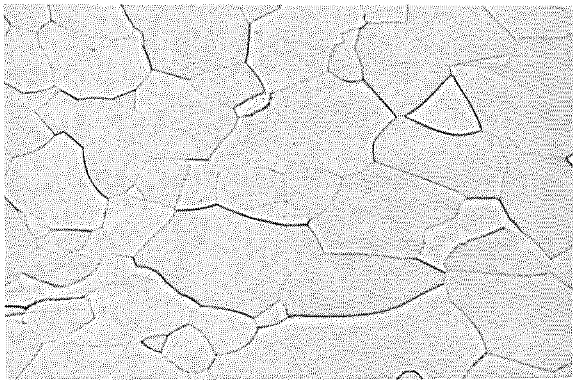
[2] Determined by the line intercept method.

material are typical for low interstitial T-111[1,2,3]. It is apparent from the data summarized in Tables 6-1 and 6-2 that the final grain size was controlled primarily by the amount of deformation received immediately prior to annealing rather than the total number of working and annealing operations. For example, the recrystallized grain size for the 4 1/4 inch diameter seamless tubing was finer than the 3 inch diameter seamless tubing as illustrated in Figure 6-3. This is not unexpected since the final reduction used in producing the 3 inch diameter tubing was relatively light (30%) as compared to the 60% final reduction for the 4 1/4 inch diameter tubing. The most significant changes in microstructure were those in the fusion zone of the welded tube shells shown in Figure 6-4. As was observed with the seamless tubing the grain size of the 3 inch tubing is larger than the 4 1/4 inch diameter tube material and the starting grain size of the welded tube shell. Thus is is apparent that the amount of work between recrystallization anneals is more critical than the initial grain size.

6.2 Determination of Post Weld and Process Annealing Temperature

The selection of the 3000°F in process and final annealing temperature was dictated primarily by two considerations. First, vacuum annealing furnaces with the required pressure and size capability were limited to 3000°F. Also, this annealing temperature has been shown to produce an optimum combination of low temperature ductility and elevated temperature creep strength in T-111. Results of post weld annealing studies had shown that 1 hour at 3000°F resulted in some homogenization of the cast metal zone, but elimination of the cellular structure did not occur until after heating for 1 hour at 3600°F (See Figures 6-5 and 6-6).

The post weld annealed samples were reduced by rolling (simulating the tube reduction schedule) and annealed for 1 hour at 2600-3000°F. Although recrystallization occurred as low as 2600°F, elimination of the evidence of the weld zone was accomplished using the 3000°F in process annealing treatment after a post weld anneal of 3000°F or 3600°F (See Figures 6-7 and 6-8). However, no significant advantage of using 3600°F over a 3000°F post weld anneal was observed. Neither was the recyrstallized grain size significantly finer for samples annealed below 3000°F. Thus the suitability of the 3000°F post weld and in process anneal was rather fortuitous in view of the equipment limitations noted earlier.

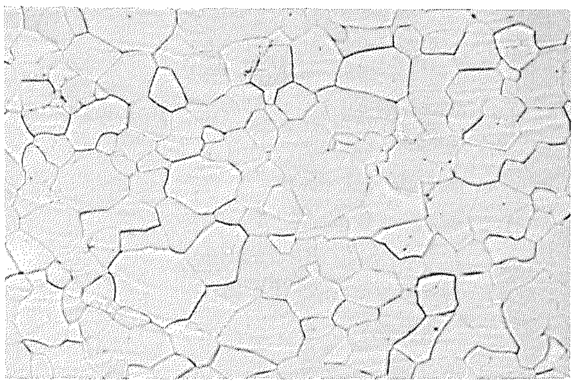


20,578B 100X

STARTING TUBE SHELL "S"

5.765" OD x .570" Wall

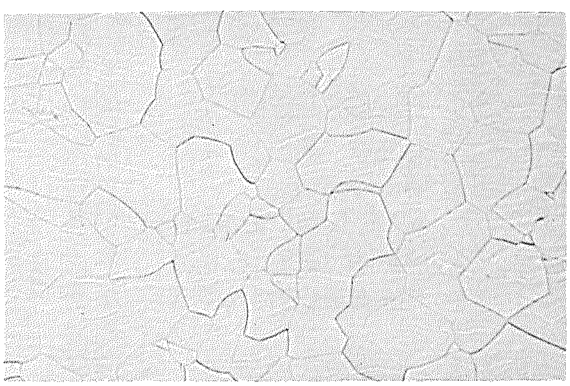
ASTM G. S. 4 to 5



20,883 100X

4 1/4" OD TUBING

4.125" OD x .135" Wall
81% Total Reduction
ASTM G.S. 6 to 7
Hardness 202 DPH

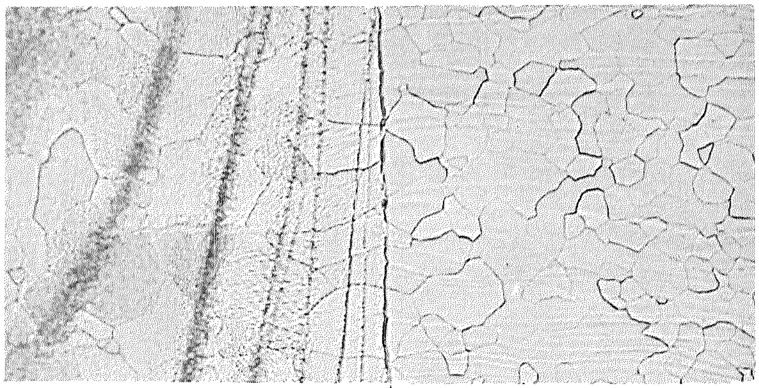


21,165 100X

3" OD TUBING

3.000" OD x .080" Wall
92% Total Reduction
ASTM G.S. 5 to 6
Hardness 199 DPH

Figure 6-3. Transverse Sections Showing Microstructure Development of 4 1/4" OD and 3" OD Seamless Tubing

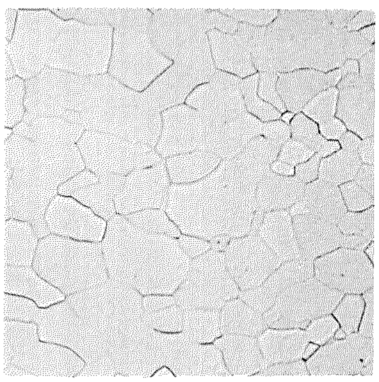


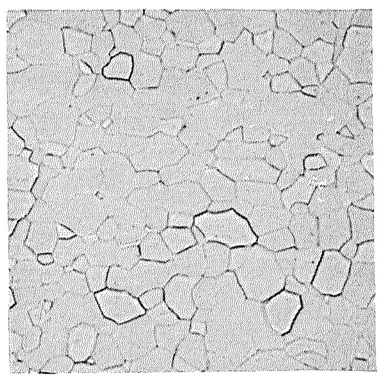
20,217 ▲ 100X
Weld Interface

AS WELDED TUBE SHELL B

5.507" OD x .425" Wall

ASTM G.S.
Weld - 4 to 5
Base - 7 to 8

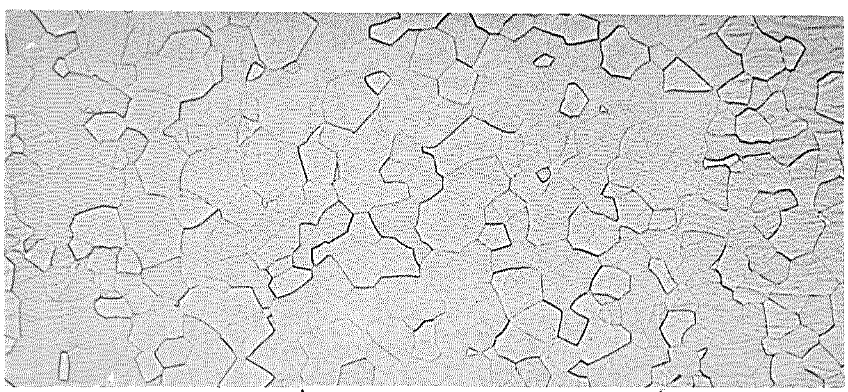




20,885 100X 20,885
Weld Base Metal

4 1/4" OD TUBING

Annealed (.125" Wall)
ASTM G.S.
Weld - 5 to 6
Base - 6 to 7
74% Total Reduction
Hardness 204 DPH
Tube B

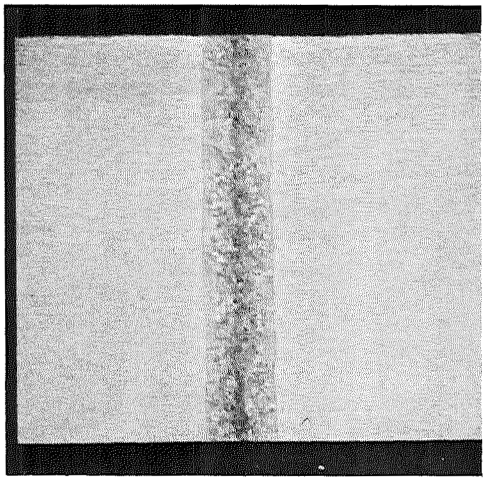


21,175 Weld 100X

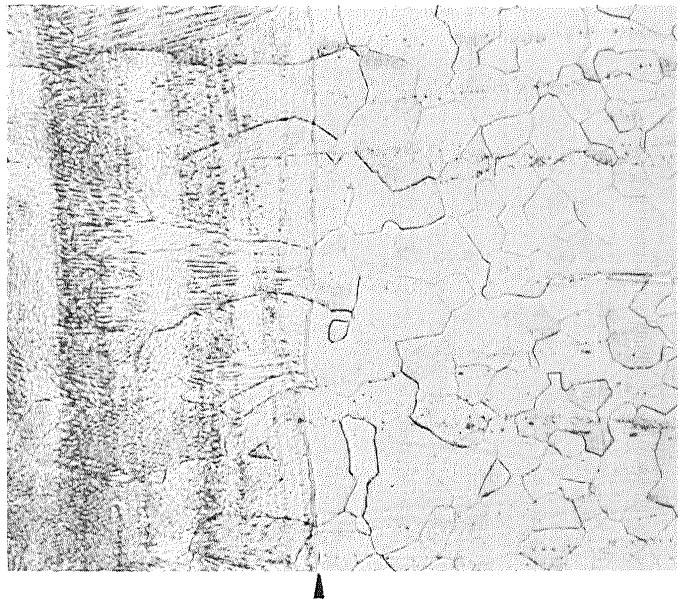
3" OD TUBING

(.080" Wall)
Annealed
87% Total Reduction
ASTM G.S. 6 to 7
Hardness 211 DPH

Figure 6-4. Transverse Sections Showing Microstructure Development of 4 1/4" OD and 3" OD Tubing Made from Welded Tube Shells

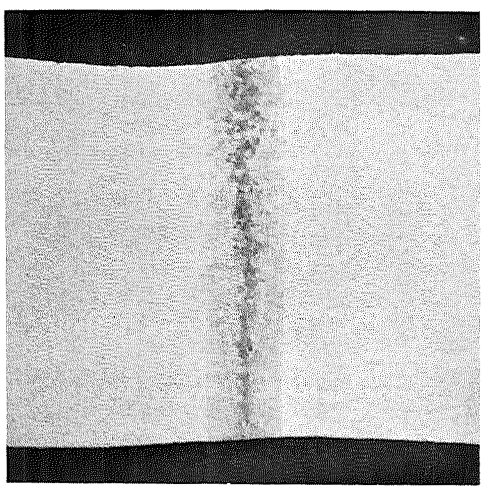


5X 19,943

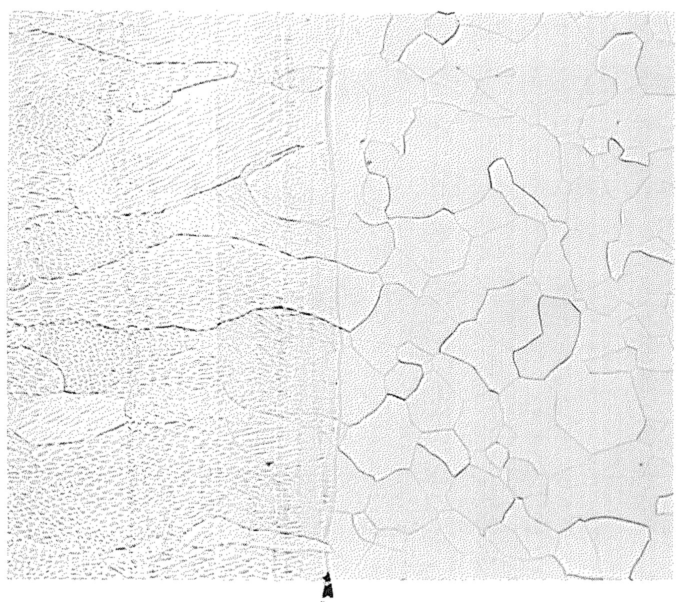


100X Weld Fusion Line 19.943

AS-WELDED



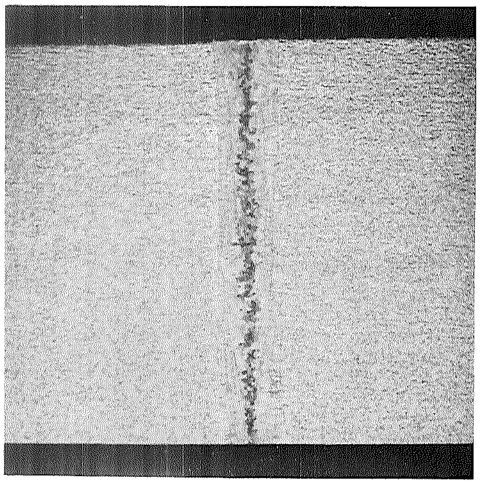
5X 19,944

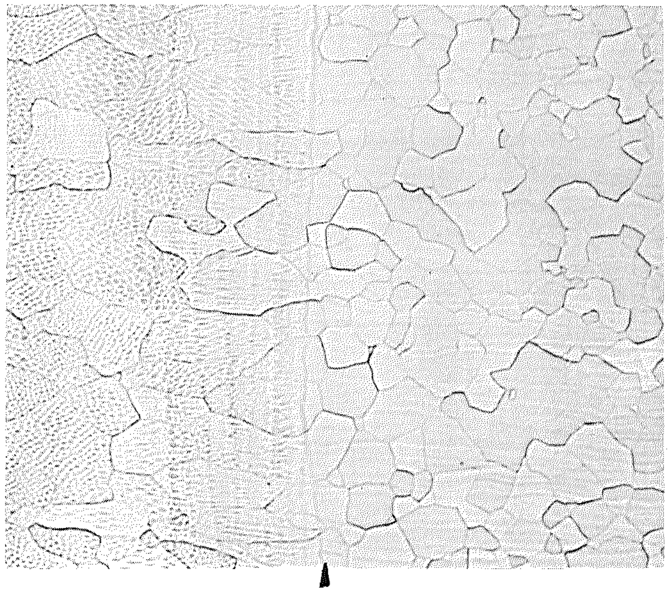


100X Weld Fusion Line 19,944

3000°F POST WELD ANNEAL

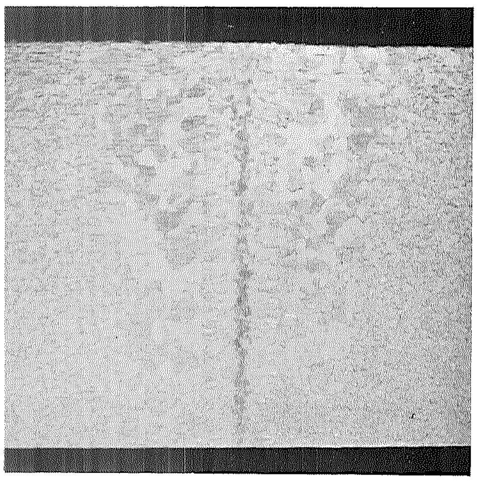
Figure 6-5. Electron Beam Weld Transverse Sections As Welded and Annealed 1 Hour at 3000°F (Plate Specimens)

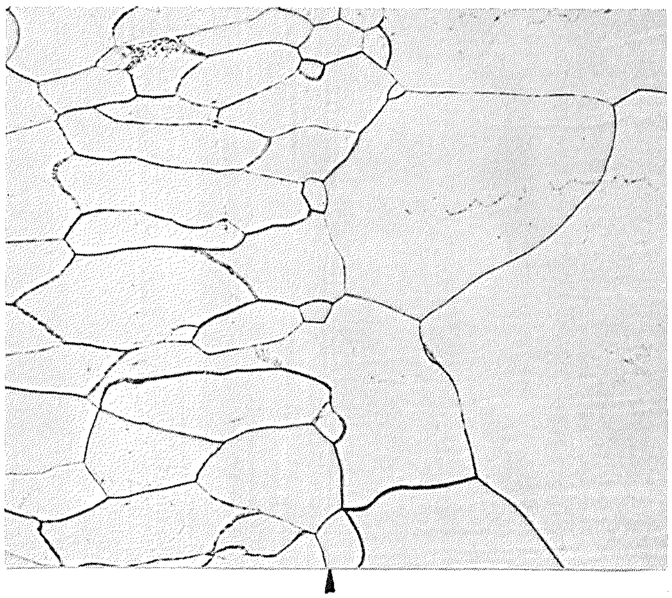




5X 19,945 100X Weld Fusion Line 19,945

3300°F POST WELD ANNEAL

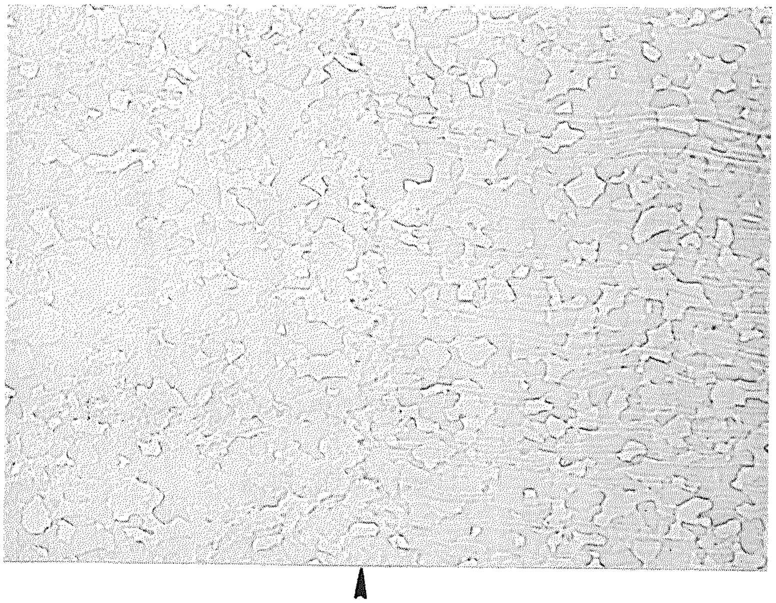




5X 19,946 100X Weld Fusion Line 19,946

3600°F POST WELD ANNEAL

Figure 6-6. Electron Beam Weld Transverse Sections Annealed for 1 Hour at 3300°F and 3600°F (Plate Specimens)

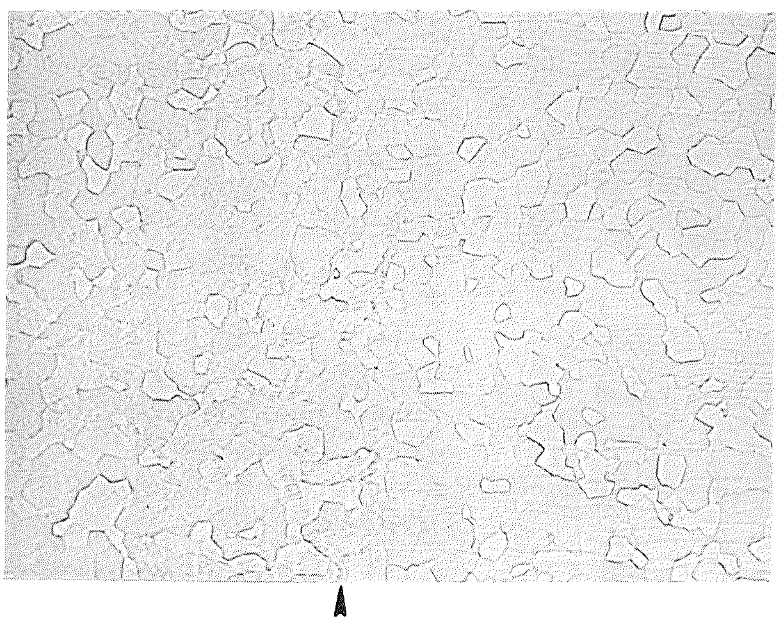


20,116 100X

Weld Fusion Line

3000°F Post Weld Anneal
Roll 44%
Anneal 1 hr. at 2600°F
Roll 56%
Anneal 1 hr. at 2600°F
Roll 35%
Anneal 1 hr. at 2600°F
Roll 35%
Anneal 1 hr. at 2600°F

ASTM G.S. 6-7



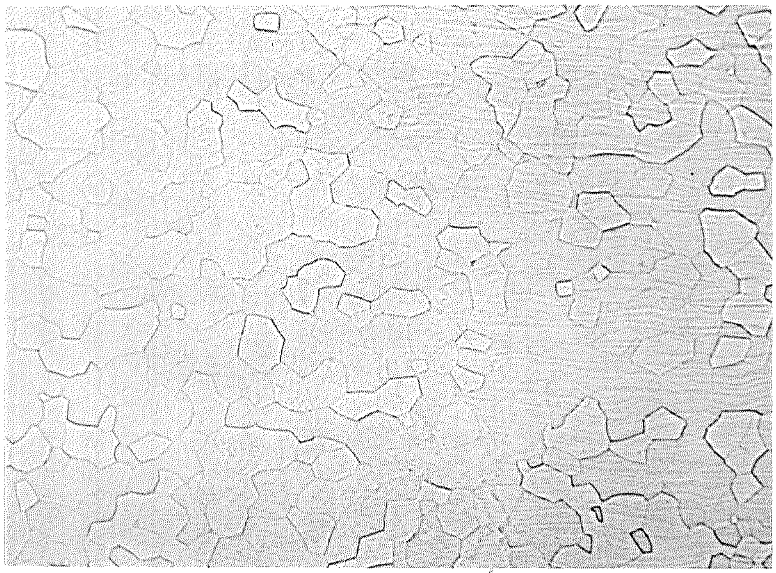
20,120 100X

Weld Fusion Line

3000°F Post Weld Anneal
Roll 44%
Anneal 1 hr. at 2800°F
Roll 56%
Anneal 1 hr. at 2800°F
Roll 35%
Anneal 1 hr. at 2800°F
Roll 35%
Anneal 1 hr. at 2800°F

ASTM G.S. 6-7

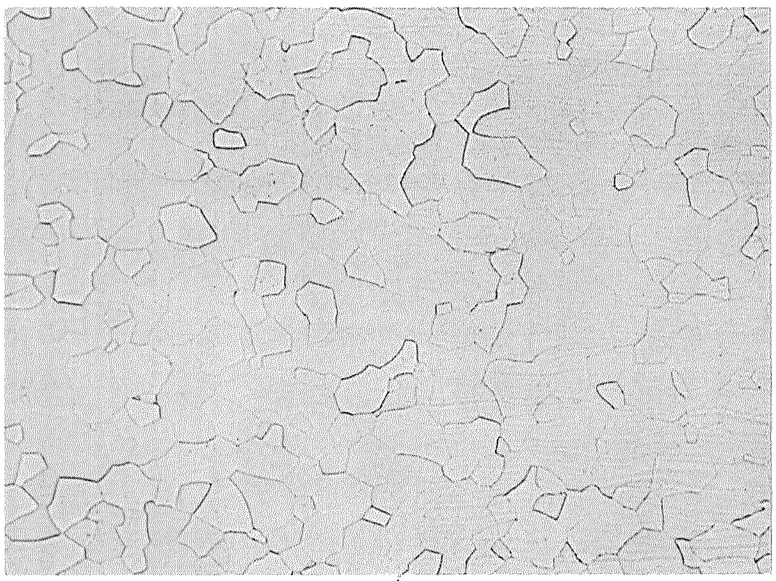
Figure 6-7. Transverse Sections of 90% Reduced Electron Beam Welds Comparing 2600°F and 2800°F Process Annealing Temperature (Plate Specimens)



20,124 Weld Fusion Line 100X

3000°F Post Weld Anneal

ASTM G. S. 6



20,126 Weld Fusion Line 100X

3600°F Post Weld Anneal

Both Specimens Processed as follows:

Roll 44%
Anneal 1 hr. 3000°F
Roll 56%
Anneal 1 hr. 3000°F
Roll 35%
Anneal 1 hr. 3000°F
Roll 35%
Anneal 1 hr. 3000°F
Total Reduction 90%

ASTM G.S. 5-6

Figure 6-8. Transverse Sections of 90% Reduced Electron Beam Welds Comparing 3000°F and 3600°F Post Weld Anneal (Plate Specimens)

6.3 Mechanical Property Evaluation

Room temperature tensile strength and 2400°F creep-rupture behavior were the mechanical properties evaluated. A pin loaded specimen with a one inch gage length and 1/4 inch width of the type shown in Figure 6-9 was used for both types of tests. Specimen length was parallel to the reduction direction for all specimens. Specimens from the welded and re-drawn tubing were sectioned to center the weld zone parallel to the specimen length. Full wall thickness specimens were used for all room temperature tensile tests and for the 2400°F creep rupture specimens taken from the 3 inch diameter tubing. Creep-rupture specimens from the 4 1/4 inch diameter x 1/8 inch thick wall tubing were ground to 0.050 inches thick by removing equal amounts from each surface. Specimens tested at full wall thickness had the tube curvature and thus the ends required flattening so they could be accommodated in the grips.

Creep Rupture

Creep-rupture testing was done in dead weight loaded, sputter ion pumped, ultra high vacuum ($< 5 \times 10^{-8}$ torr) system of the type described by Buckman and Hetherington[6]. Creep strain was measured optically by directly observing the separation of fiducial marks applied to the specimen gage length. Supplementing the optical strain measurement (which was limited to 40% extension) was a dial gage monitoring the load strain displacement.

The 2400°F creep-rupture properties for each size and type tubing is summarized in Table 6-3 and presented graphically in Figure 6-10. As shown in Figure 6-10, the stress rupture behavior of the T-111 tubing agrees reasonably well with the short time rupture data for T-111 obtained at 10^{-5} to 10^{-6} torr. The time to 1% strain data is also shown in Figure 6-10 and compares well to lower stress level data previously reported for T-111.[7] All the data shown in Figure 6-10 was for material that had been annealed for 1 hour at 3000°F prior to test. Creep curves observed for the T-111 specimens typically consisted of essentially no loading strain and then a continuously increasing creep rate as shown in Figure 6-11. Rupture elongation for all the specimens tested was in the range of 51-88% and reduction in area values were

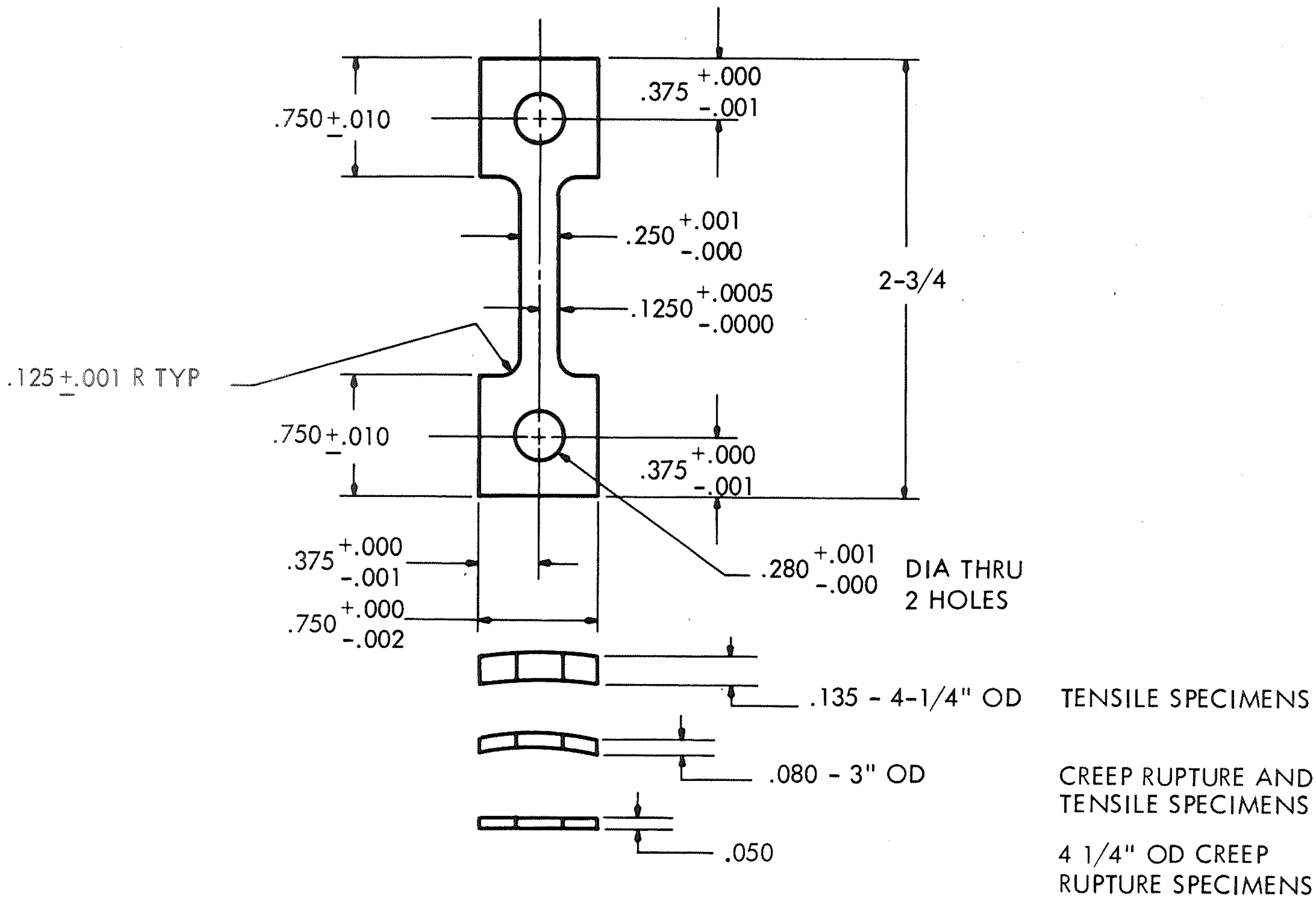


Figure 6-9. **Tensile and Creep Rupture Specimen Configuration**

Table 6-3. 2400°F Creep Rupture Test Results for T-111 Tubing

Spec. No.	Description	Stress psi	Time to Rupture Hrs.	Time to 0.5% Strain Hrs.	Time to 1.0% Strain Hrs.	Reduction in Area %	Elongation %	ASTM Grain Size
			4 1/4 inch diameter tubing*					
B-1B-6	Welded tube Base metal	28,000	2.1	---	---	69	51	6-7
B-1B-4	Welded tube Base metal	20,000	45.1	0.3	1.1	66	81	6-7
S-2A-4	Seamless Tube	18,000	52.0	0.5	1.5	66	55	6-7
B-1W-6	Welded tube Long. weld	18,000	67.7	1.0	3.0	75	74	5-6
B-1B-5	Welded tube Base metal	16,000	199.7	7.0	16.0	77	67	6-7
			3 inch diameter tubing**					
C-2B-4	Welded tube Base metal	18,000	92.7	2.5	6.0	79	74	6-7
C-2W-4	Welded tube Long. weld	18,000	95.1	2.5	5.5	78	73	6-7
S-1B-4	Seamless Tube	18,000	99.5	2.0	4.5	72	88	4-5

*4 1/4" OD specimens ground flat to .050" thickness.
**3" OD specimens tested at .080" wall thickness and 1 1/2" radius.

Note: All tests conducted at $< 5 \times 10^{-8}$ torr.

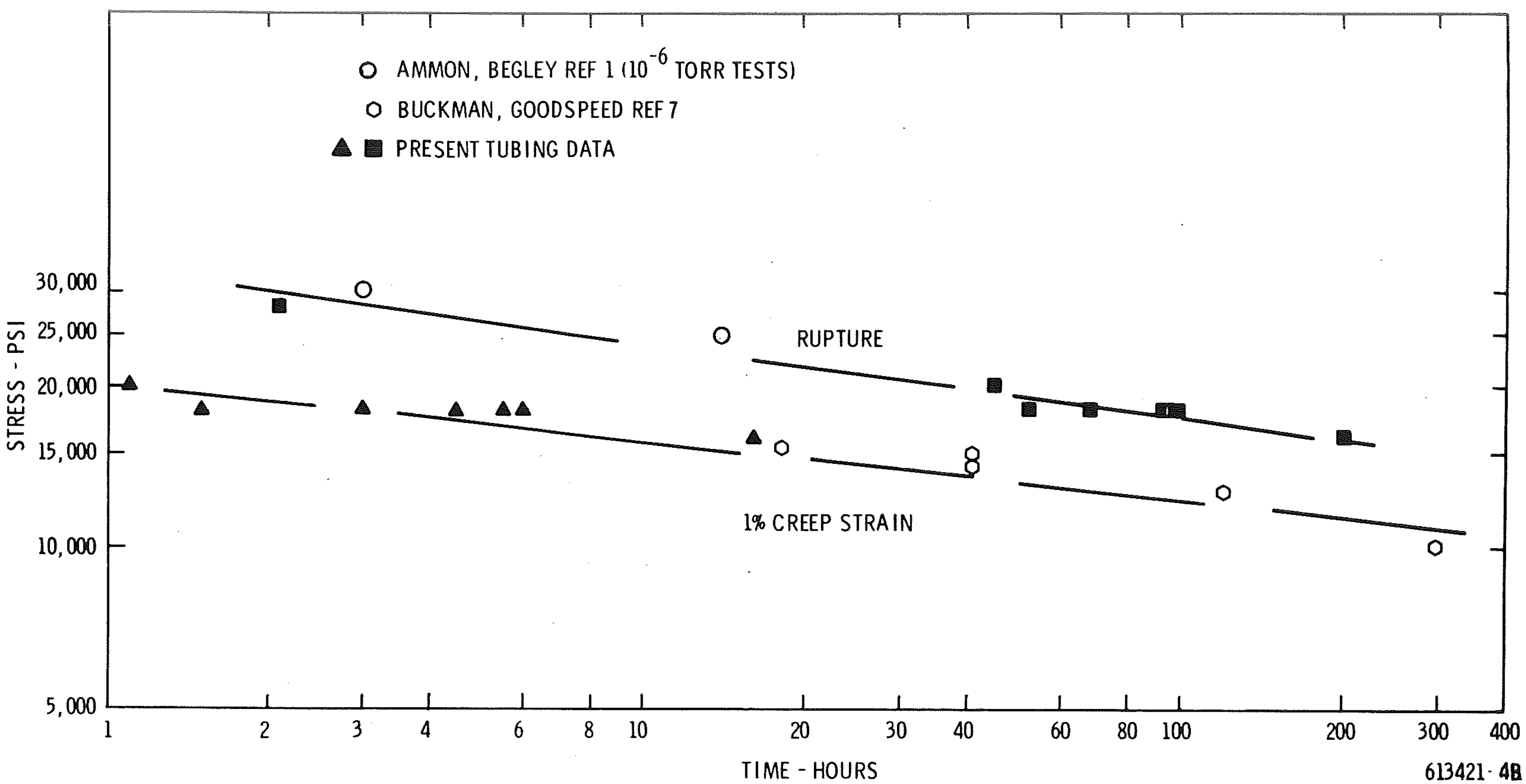


Figure 6-10. 2400°F Creep Rupture Strength of T-111 Tubing

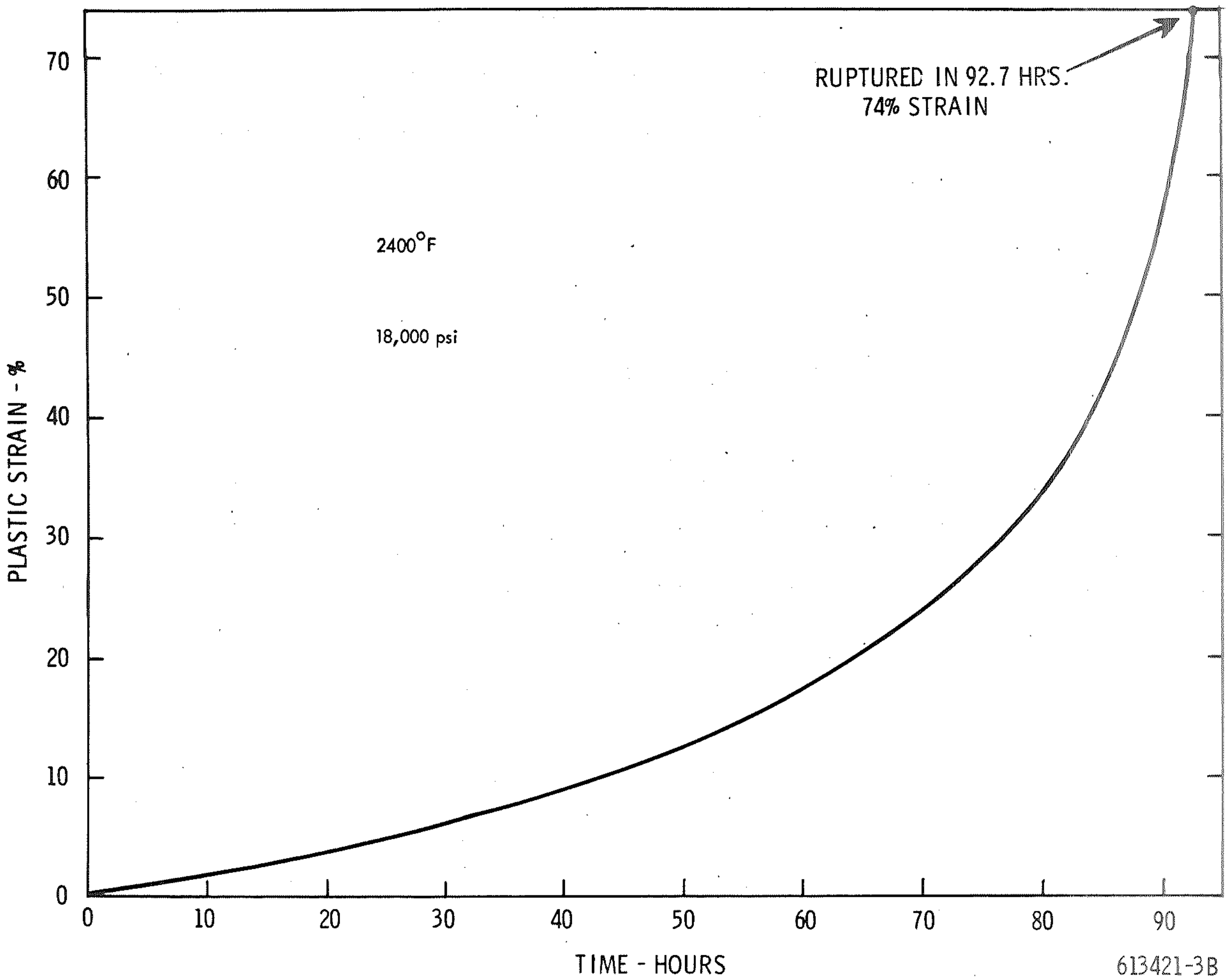


Figure 6-11. Typical Creep Rupture Curve

70-80%. Fracture occurred primarily by transgranular shear as shown in the Figures 6-12, 6-13, and 6-14. Although some intergranular separation is evident, the serrated grain boundaries indicate boundary mobility.

The 2400°F creep rupture behavior of the seamless and welded-redrawn T-111 tubing was essentially identical and neither did there appear to be any size effect. The creep behavior also did not significantly differ from that reported for T-111 sheet of comparable grain size and thermal history.

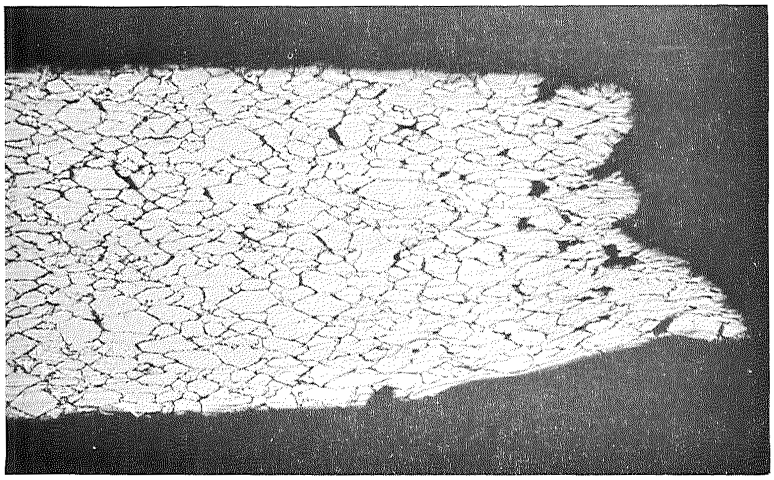
Tensile Properties

Full wall thickness, longitudinal tensile specimens were tested in triplicate at room temperature for each size of welded and seamless tubing. As shown by the results presented in Table 6-4, there was no significant difference between either size or type of tubing. The properties are typical for recrystallized T-111 as shown by the comparative data in Table 6-5. Excellent ductility was exhibited by all specimens and fractures were typically by transgranular shear after a high reduction in area (See Figure 6-15).

6.4 Chemical Analysis of Tubing

The high reactivity of T-111, typical of refractory metal alloys, requires careful monitoring of the chemical composition to ensure no significant changes occur during the various processing operations. This is a necessity since extraneous contamination can significantly alter the processing characteristics and final mechanical properties of T-111.

Strict controls over each stage of processing from ingot consolidation to the final annealing operation resulted in essentially no significant changes in composition of the T-111 material that could be attributed to extraneous cause as shown by the analytical data tabulated in Tables 6-6 and 6-7. Although there are some minor variations in the carbon, oxygen, and nitrogen values, these are most likely attributable to heterogeneous distribution and the

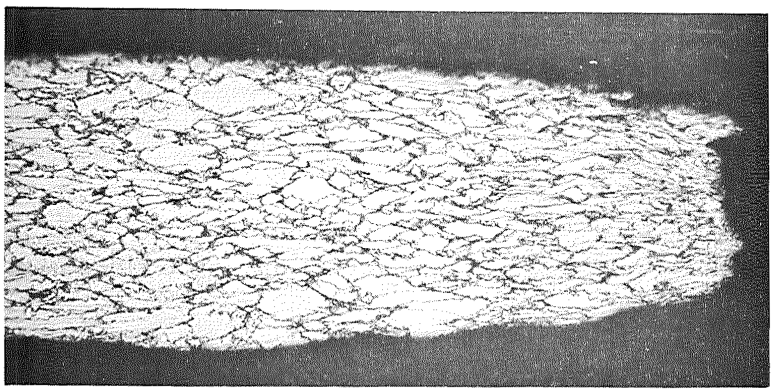


50X 21,276

2.1 hrs. Rupture Time
28,000 psi 51% Elongation

4 1/4" OD x .135" Wall
Specimen .050" Thick

B-1B-6
Welded Tube B
Base Metal Section

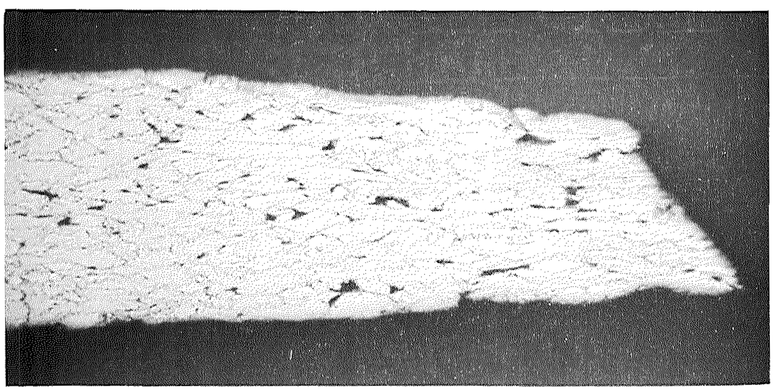


50X 21,275

45 hrs. Rupture Time
20,000 psi 81% Elongation

4 1/4" OD x .135" Wall
Specimen .050" Thick

B-1B-4
Welded Tube B
Base Metal Section



50X 21,273

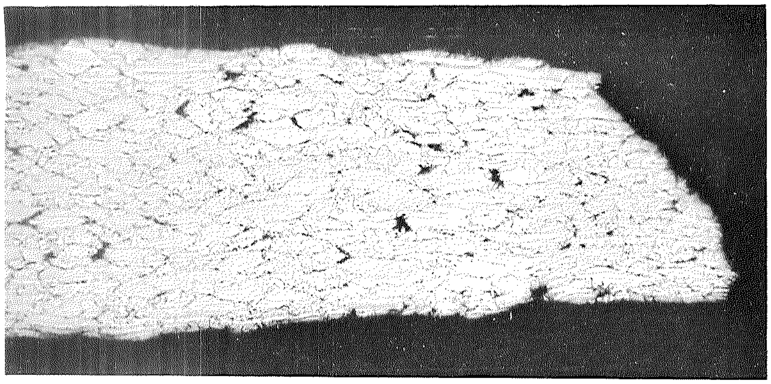
.050" Thick Flat Ground Specimens

68 hrs. Rupture Time
18,000 psi 74% Elongation

4 1/4" OD x .135" Wall
Specimen .050" Thick

B-1W-6
Welded Tube B
Longitudinal Weld Section

Figure 6-12. 2400°F 4 1/4" OD Tubing Creep Rupture Fractures

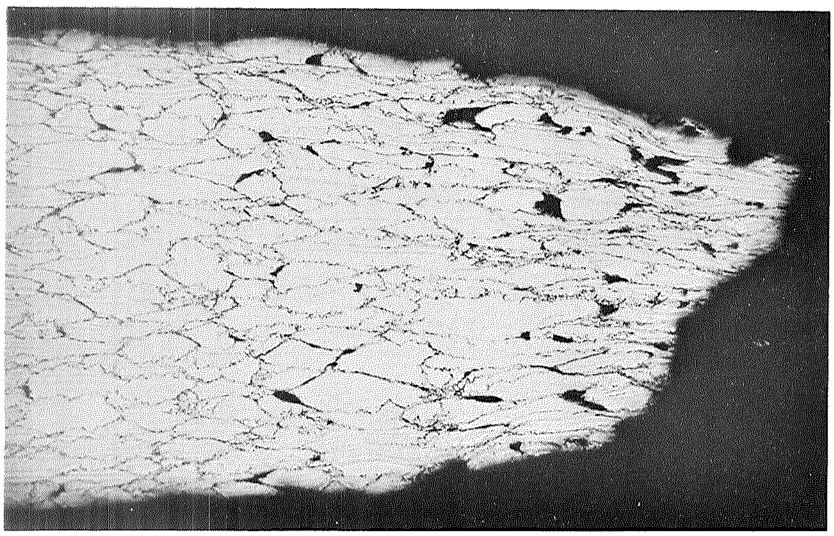


52 hrs. Rupture Time
18,000 psi 55% Elongation

4 1/4" OD x .135" Wall
Ground to .050" Thickness

S-2A-4
Seamless Tube

50X 21,274



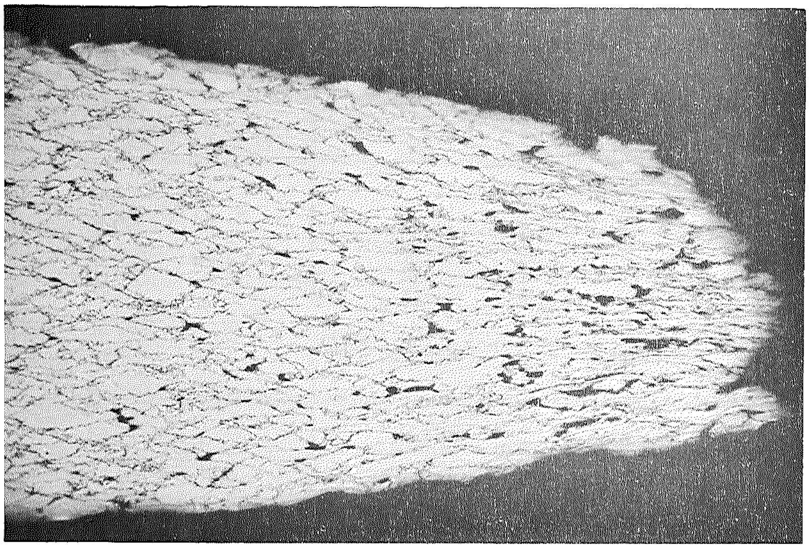
100 hrs. Rupture Time
18,000 psi 88% Elongation

3" OD x .080" Wall
Specimen .080" Thick

S-1B-4
Seamless Tube

50X 21,795

Figure 6-13. 2400°F 4 1/4" OD and 3" OD Seamless Tubing Creep Rupture Fractures

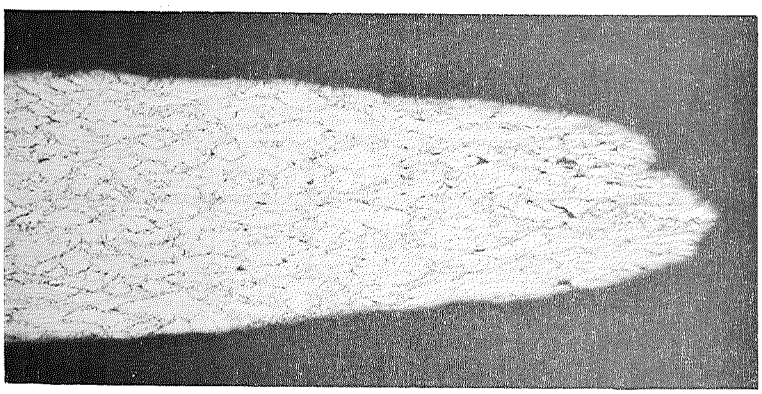


95 hrs. Rupture Time
18,000 psi 73% Elongation

3" OD x .080" Wall
Specimen .080" Thick

C-2W-4
Welded Tube C
Longitudinal Weld Section

50X a. 21,797



199 hrs. Rupture Time
16,000 psi 67% Elongation

4 1/4" OD x .135" Wall
Specimen .050" Thick

B-1B-5
Welded Tube
Base Metal Section

50X b. 21,272

Figure 6-14. 2400°F 4 1/4" OD and 3" OD Welded Tubing Creep Rupture Fractures

Table 6-4. Room Temperature Tensile Properties of T-111 Tubing*

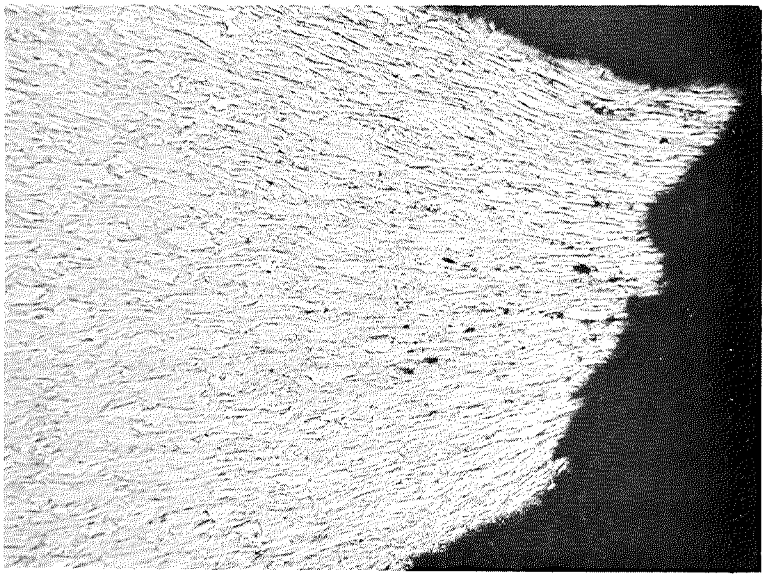
Specimen	UTS (KSI)	0.2% Y.S. (KSI)	Elongation (%)	Tubing Diameter
	Welded Tubing Weld Zone			
B-1W-2	89.3	77.0	40.8	4 1/4" OD .135" wall
B-1W-3	89.6	75.8	40.4	
B-1W-5	89.7	75.4	42.5	
Ave.	89.5	76.1	41.2	
σ	.23	.83	1.12	
C-2W-1	90.8	72.6	37.08	3" OD .080" wall
C-2W-2	90.9	73.6	37.60	
C-2W-3	90.8	75.0	36.88	
Ave.	90.8	73.7	36.2	
σ	.06	1.21	1.86	
	Welded Tubing Base Metal			
B-1B-1	89.7	77.8	38.5	4 1/4" OD .135" wall
B-1B-2	89.5	74.5	37.3	
B-1B-3	89.8	76.3	40.6	
Ave.	89.7	76.2	38.8	
σ	.15	1.65	1.67	
C-2B-1	91.6	73.4	35.73	3" OD .080" wall
C-2B-2	91.4	74.0	36.28	
C-2B-3	91.2	74.4	35.25	
Ave.	91.4	73.9	35.8	
σ	0.20	0.51	0.52	
	Seamless Tubing			
S-2A-1	86.7	72.6	39.9	4 1/4" OD .135" wall
S-2A-2	86.8	72.1	41.2	
S-2A-3	86.3	71.4	39.2	
Ave.	86.6	72.0	40.1	
σ	.29	.60	1.01	
S-1B-1	87.8	71.3	35.38	3" OD .080" wall
S-1B-2	88.6	71.5	38.65	
S-1B-3	88.4	70.2	34.70	
Ave.	88.3	71.0	36.2	
σ	0.42	0.70	2.11	

*Full wall thickness specimens with specimen axis parallel to working direction tested at 0.05 in/min.

Table 6-5. Room Temperature Tensile Properties of T-111

Source	UTS ksi	0.2% Y.S. ksi	Elongation %	Strain Rate* in/in/min
Tube B-Weld Long.	89.5	76.1	41	05
Tube B-Base Metal	89.7	76.2	39	.05
Tube C-Weld Long.	90.8	73.7	36	.05
Tube C-Base Metal	91.4	73.9	36	.05
4 1/4" OD Seamless	86.6	72.0	40	.05
3" OD Seamless	88.3	71.0	36	.05
Cold rolled 90%(1) 1 hr 3000°F	90.4	82.5	29.0	.005
.035" Sheet (4)	89.2	83.2	16.0	.005
Transverse Weld in .035" Sheet Post (4) Weld annealed at 2400°F	92.0		14.0	.005

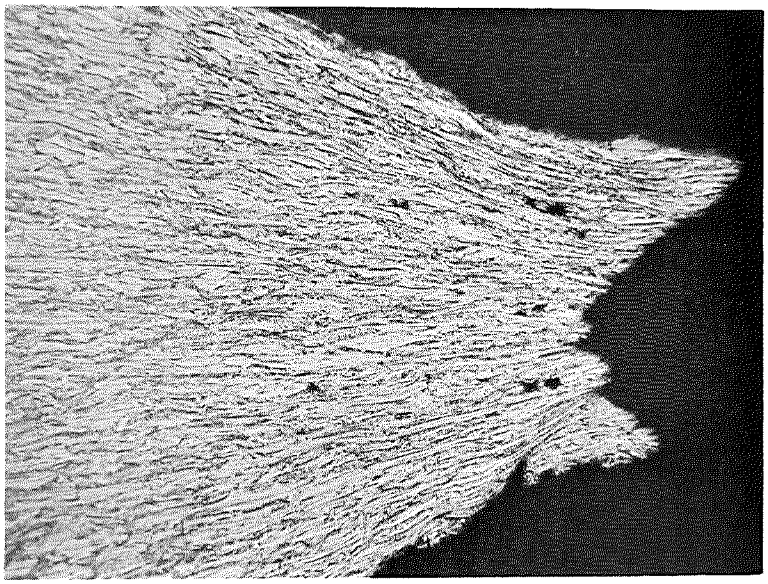
*Based on Cross Head Travel



Tube B-1W-2
Welded Tube B
Longitudinal Weld Section

89,300 psi U.T.S.
77,000 psi Y.S.
41% Elongation

50X 21,278



Tube S-2A-3
Seamless Tube S

86,300 psi U.T.S.
71,400 psi Y.S.
39% Elongation

50X 21,279

Full wall thickness, curved specimens.

Figure 6-15. 4 1/4" OD Room Temperature Tensile Fractures

Table 6-6. Chemical Analysis of Seamless and Weld-Reduced Tubing

	w/o		ppm by weight			
Sample Source	W	Hf	C	O	N	H
Seamless Tubing as Cast Ingot	8.1	1.86	14	18	12	0.6
As Extruded			59	24	16	4.4
Annealed				25		
4 1/4" S-2A Annealed	8.2	1.85	25	20	<2	0.5
5 3/4" T Annealed				25		
3" S-1B-F			44	22	<10	
As Cast Ingot for Welded Tubing	8.7	2.0	33	23	10	1.0
As Extruded			34	38	9	
Annealed			45	38	3	
Sheet Bar A Annealed			48	25	29	0.7
Rolled Plate			48	51	20	1.2
Annealed Plate			40	40	40	1.0
5" Long Cylinder P as Annealed			41	26	<5	1.7
Electron Beam Weld Metal	8.3	2.1	22	31	40	6.7
Pipe C 5 1/2" OD				45		
Pipe B 5" OD				27		
Pipe C-2 3"			24	26	<10	
2400°F Creep Rupture Specimens						
B-1B-6 6 hr.			10	41	<10	
B-1W-6 67 hr.			50	40	<2	
B-1B-5 200 hr.			20	44	<10	

Table 6-7. Spectrochemical Analyses for Trace Impurities in T-111 Alloy Tubing

	K	Li	Na	B	Si	Fe	Pb	Cr	Mg	Cb	Mn	Ni	Al	Sn	V	Cu	Mo	Ti	Ca	Zr	Co	Zr
Ingot for Seamless Tubing					<60	<60		<60		1000	40	<60	40		<20	20	60	<20			<60	
Ingot for Welded Tubing Vendor Analyses					<20	20		<10		350		<10	<10		<10	<20	15	<20			<5	
S-2A 4 1/4" OD Seamless Tube	<5	<5	<5	<3	<10	<10	<3	<3	<3	<100	<3	<10	<3	<3	<10	<20	<30	<10	<3	<30	<3	200/1000*

*Hafnium contains 2.5% Zirconium.

intrinsic difficulties associated with interstitial analysis. The major alloy additions and trace impurities were within the specification limits presented in Table 6-8, and the trace and interstitial levels were maintained well below the maximum throughout the entire processing sequence.

To lessen the influence of sample preparation on the analytical results, all samples for carbon, oxygen, nitrogen, and hydrogen were 1/8 inch cubes prepared by low speed cutting operations and were hand filed and lightly etched prior to analysis (See Table 6-9). This is important because most trace contaminants are surface contaminants and thus influence the impurity analysis in relation to the surface to volume ratio of the specimen.

Table 6-8. T-111 Chemical Composition Specification

Ingot Requirements		
Element	Minimum Content (ppm)	Maximum Content (ppm)
Carbon	---	50
Nitrogen	---	50
Oxygen	---	100
Hydrogen	---	10
Columbium	---	1000
Molybdenum	---	200
Nickel	---	50
Copper	---	50
Cobalt	---	50
Iron	---	50
Vanadium	---	20
Tungsten	7.0 w/o	9.0 w/o
Hafnium	1.8 w/o	2.4 w/o
Tantalum	Remainder	

The final as-melted ingot was sampled at the center, mid-radius and edge at the top and bottom and at the edge in the center and analyzed for W, Hf, C, O, N, and H.

Rolled Plate Interstitial Requirements		
Element	Max, ppm	Permissible Variation in Check Analysis, ppm
C	50	+10
O	150	+20
N	75	+10
H	10	+2

Table 6-9. Chemical Analysis Techniques Employed

Element	Technique	Specimen Size
Oxygen	Vacuum fusion	1/8" cubes placed in molten Pt
Nitrogen	Micro Kehldahl	1/8" cubes, distillation
Carbon	"Leco" low carbon analyser	1/8" cubes or chips - oxidized
Hydrogen	Mass spectrometer	1/8" cubes
Tungsten Hafnium	X-ray fluorescence	2 gram specimen
Trace Impurities	Spectrographic	1/8" cubes

7. CONCLUSIONS

Using current commercial metal working equipment, and by exercising strict control over each stage of processing, high quality large diameter T-111 tubing can be produced from either seamless or welded tube shells.

Because of the extra fabrication steps required to produce large sections of .4 inch thick plate for the welded tube shell, the seamless tubing was fabricated with considerably less difficulty than the weld-reduced tubing. An overall product yield from ingot to completed tubing of 41% was obtained for the seamless tubing as compared to 30% for the weld reduced tubing. The ready availability of T-111 plate stock or a requirement for thinner walled tube shells could alter the cost comparison between the two types of tubing.

The inventory of finished tubing, following sectioning for evaluation specimens is listed below:

Tube No.	Description	Length	Diameter	Weight	Condition
B-1	Weld-Reduced	90"	4 1/4" OD x .125" wall	88 lbs	Annealed
S-2	Seamless	120"	4 1/4" OD x .135" wall	118 lbs	Cold Worked
S-1B	Seamless	104"	3" OD x .080" wall	48 lbs	Cold Worked
S-1A	Seamless	9"	3" OD x .080" wall	4 lbs	Annealed
C-1	Weld-Reduced	77"	3" OD x .080" wall	40 lbs	Annealed
C-2	Weld-Reduced	83"	3" OD x .080" wall	45 lbs	Annealed

Since vacuum annealing required sectioning to a maximum length of 85", seamless tubes S-2 and S-1B were not annealed to retain the longest possible length for future application.

Lubrication Difficulties

A prerequisite to future T-111 tubing fabrication program is the development of adequate tube reducer lubricants. Conventional cold pilger lubricants did not provide reliable protection to the tube surface and dies. Experience with similar alloys indicates that a conversion coating on the tantalum alloy is required to retain the lubricant and to prevent die galling. Oxide or anodized films and a uniformly roughened surface is suggested. Solid film lubricants, provided they are acceptable to the tube reducing equipment, may also offer an advantage.

Also, the following additional conclusions can be made based on the program results.

- Heavy section, .425" thick longitudinal butt welds up to 29 inches in length can be made in T-111 tubes using single pass electron beam welding.
- Single pass, .070" wide electron beam welds can be satisfactorily reduced 90% in area with appropriate intermediate anneals, to provide a completely homogenized, fine grained structure.
- Evacuated mild steel containers provided adequate protection for the T-111 billets during the 2300°F extrusion operation.
- Open air forging from a 2300°F gas fired furnace can be accomplished without excessive contamination or scaling loss by using a aluminum - 12 w/o silicon billet coating.
- Cold pilger tube reduction passes as high as 65% can be accommodated by T-111 without material or equipment difficulty.

Based on the experience gained in this program, the following example is presented showing the best estimation for producing 5 1/2 inch OD and 4 inch OD T-111 tubing. In our estimation, a single reduction pass from tube shell to finished tubing would promise better results than the two passes used in this program. The sizes considered are within the capabilities of the Canton Drop Forge extrusion press and Timken's 6 1/2 inch maximum OD cold pilger machine. The same calculations may be applied to smaller diameter finished tubing or larger sized assuming larger sizes and capacities in ingot melting, (over 10" diameter) in extrusion press capacity (5500 ton rating, 3700 ton safe limit) and in tube rocking or drawing capacity (over 6 1/2 inch diameter). Table 7-1 shows the assumption used and Table 7-2 shows sample calculations for 4 inch OD x 1/8 inch wall and 5 1/2 inch OD x 1/8 inch wall tubing. The maximum size of seamless tubing that could be made using Timken's pilger mill would appear to be 5 1/2 inches diameter.

Table 7-1. Dimensional Relationships for Hollow T-111 Extrusion

- The following dimensional review begins with the machined tube shell and works in reverse to the extrusion and billet dimensions.

- To determine as extruded T-111 dimensions, add .150" machining allowance per surface.

- To determine T-111 extrusion billet ID, add .300" to declad extrusion ID.

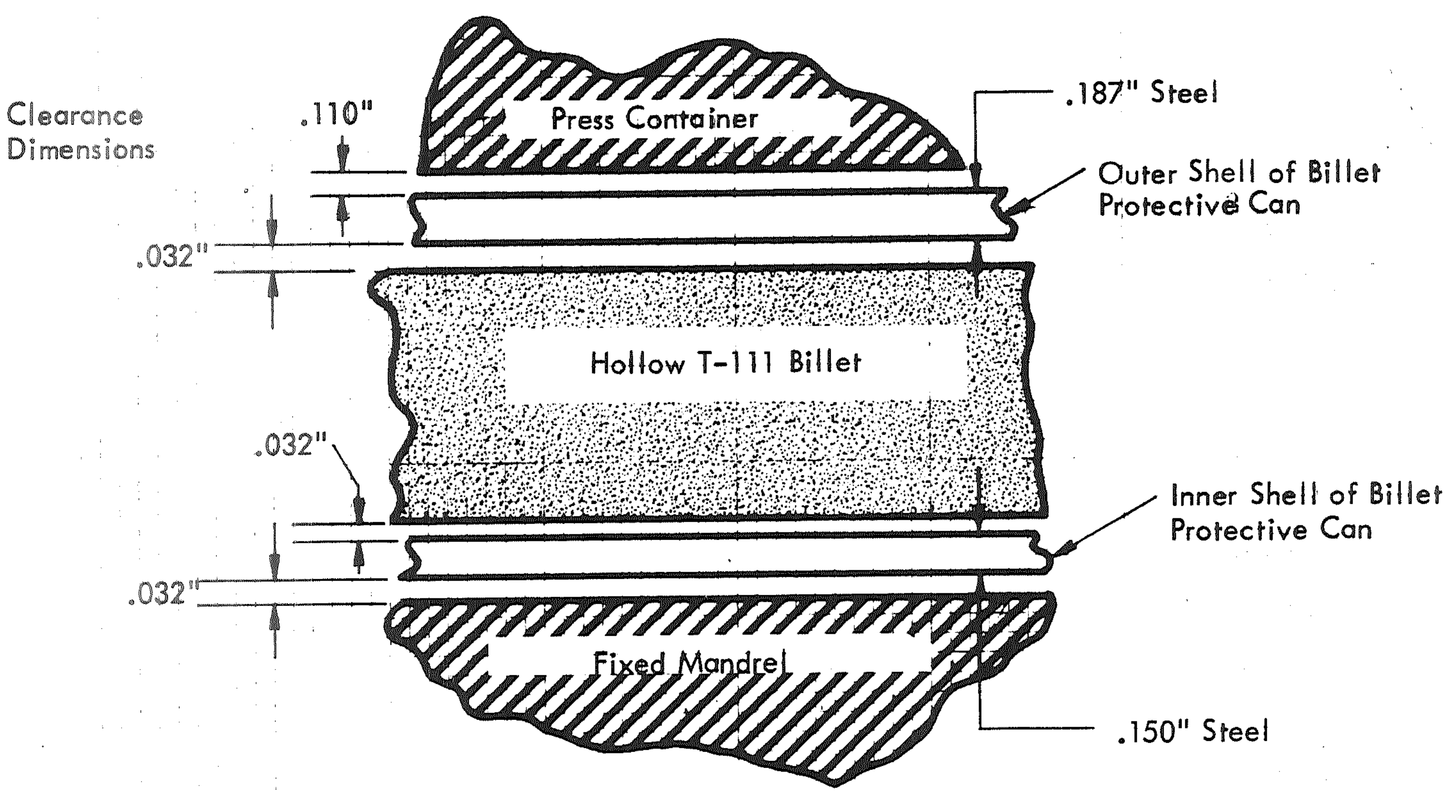
- Extrusion billet OD is determined by extrusion ratio desired, available ingot size and press capacity.

- To determine the press container ID, add .700" to T-111 billet OD.

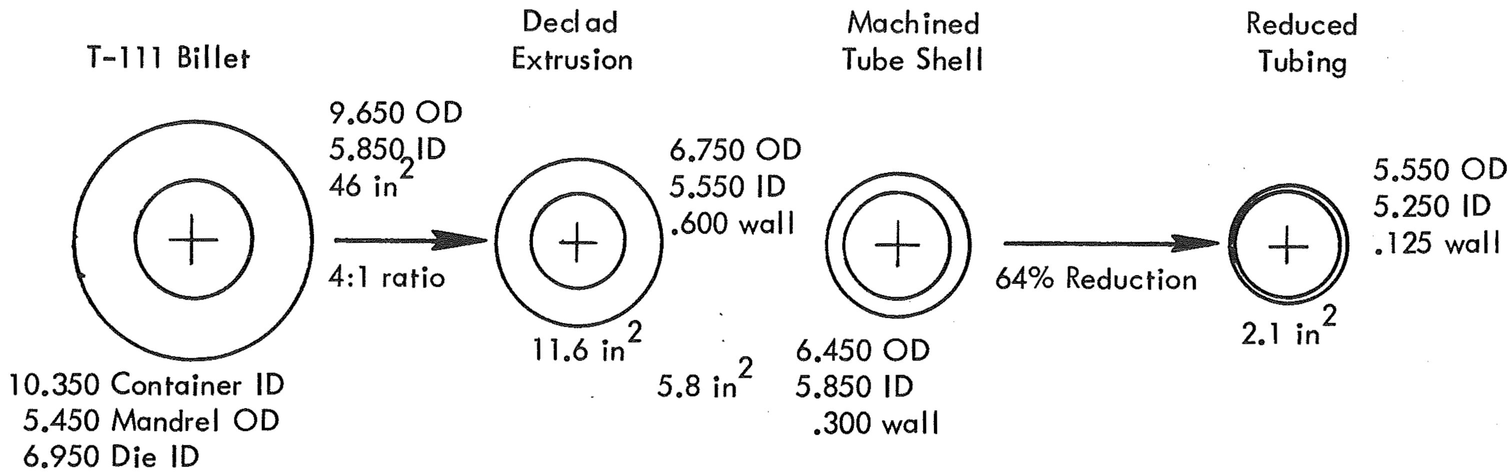
- To determine mandrel diameter:

  subtract .100" from declad T-111 extrusion ID
  subtract .400" from T-111 billet ID.

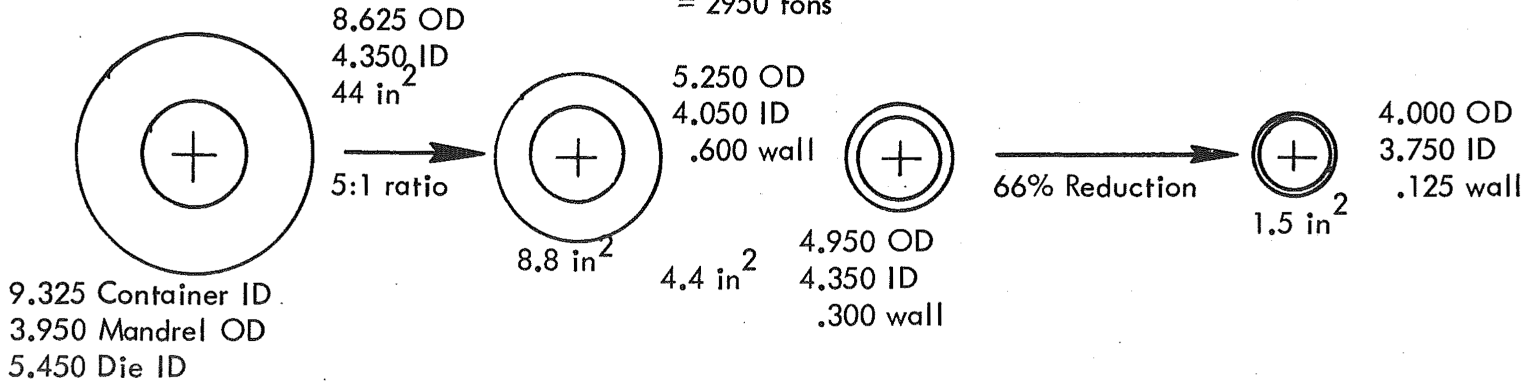
- To determine die size, add .200" to declad T-111 extrusion OD.



Longitudinal Cross Section of Hollow T-111 Billet



Extrusion Force = $K\ Ao \ln Ao/A_1$ = (40 ksi) (55 in^2) (ln 3.82)
= 2950 tons



Extrusion Force = $K\ Ao \ln Ao/A_1$ = (40 ksi) (56.4 in^2) ln 5.1
= 3700 tons

K for T-111 at 2300°F = 40 ksi
Ao = (Area Press Container) - (Area Mandrel)
A_1 = (Area Die) - (Area Mandrel)

Table 7-2. Sample Calculations for Single Pass Tube Reduction of T-111

Weld reduced tubing made from welded tube shells appears to be better suited for thin wall tube shells (less than 1/4 inch thick) destined for thin wall tubing. An additional application, of course, is to use as-welded tubing for very large diameter and very thin wall tubing which may be too difficult to produce using tube reduction or tube drawing.

TYPICAL T-111 TUBING PROPERTIES
Recrystallized 1 hour at 3000°F

Room Temperature

Tensile Strength	-	90,000 psi
Yield Strength	-	75,000 psi
Elongation	-	40%
Hardness	-	200 DPH
Grain Size	-	ASTM 6

2400°F 100 Hour Creep Rupture Strength in Vacuum

18,000 psi
75% Reduction in Area
75% Elongation

REFERENCES

1. R. L. Ammon and R. T. Begley, "Pilot Production and Evaluation of Tantalum Alloy Sheet," Summary Phase Report, WANL-PR-M-004, June 15, 1963.

2. R. W. Buckman, Jr., J. L. Godshall, "Development of High Strength Columbium and Tantalum Alloy Tubing," Final Technical Report WANL-PR-(N)-004, May 1964.

3. Anon., "Status of Refractory Alloy Tubing," 1964 Report MAB-208-M, August 16, 1965, p. 24.

4. G. G. Lessmann, "Effect of 1000 Hour Thermal Exposure on Tensile Properties of Refractory Metal Alloys," NASA-CR-72095, WANL-PR-(P)-012.

5. F. A. DeSaw et al, "Development of a Manufacturing Method for the Production of Aircraft Structural Components of Titanium by High Frequency Resistance Welding," B.M.I. Interim Report IR-817-9(1) for AFML May 1970.

6. R. W. Buckman and J. J. Hetherington, "An Apparatus for Determining Creep Behavior under Conditions of Ultra High Vacuum," Review of Scientific Instruments, Vol. 37, No. 8, August, 1961.

7. R. W. Buckman, Jr., and R. C. Goodspeed, "Development of Dispersion Strengthened Tantalum Base Alloy," 11th Quarterly Report, WANL-PR(Q)-012, NASA-CR-72094.

APPENDIX I

Subcontractor Details

APPENDIX 1

Subcontractor Details

1. Wah Chang Division Teledyne Corp.
   P. O. Box 460, Albany, Oregon 97321

   Refractory metal alloy melting. Vacuum consumable arc melting, 10" dia. x 70" length.
   2400 lbs. tantalum alloy.
   Electron beam melting.
   Surface conditioning.
   Pickling - 60 v/o water, 30 v/o HNO_3, 10 v/o HF, 85" length x 5" dia.
   Vacuum Annealing - Cold wall diffusion pumped furnace. 24" dia. x 80" heater length.
   85" maximum workpiece length. See Figure A1-1 for temperature distribution.
   3000°F maximum temperature - $\leq 5 \times 10^{-5}$ torr pressure.
   Heater and 9 heat shields are refractory metal.
   Only refractory metal alloys heat treated.
   Tantalum foil wrapping required on thin gage to provide contamination-free annealing.
   Personnel: General Marketing - G. Liadas
   Melting - W. E. Maurer
   Fabrication - M. D. McNabb
   Annealing - L. G. Findley

2. Thermo Electron
   7 Crane Court
   Woburn, Massachusetts 01801

   Electro dischrage machining.

3. The Canton Drop Forging & Manufacturing Co.
   12th Street S.W.
   Canton, Ohio 44702

   Hot extrusion.
   5500 ton Loewy Hydropress - 60" dia. piston.
   3700 tons maximum at start to avoid stalling.
   Maximum billet length - solid - 30" hollow - 15" with mandrel.
   Container size to 17".
   Salt bath heating to 2300°F.
   Personnel: Marketing - R. Swallen
   Technical - F. Welshner
   Foreman - P. Moore

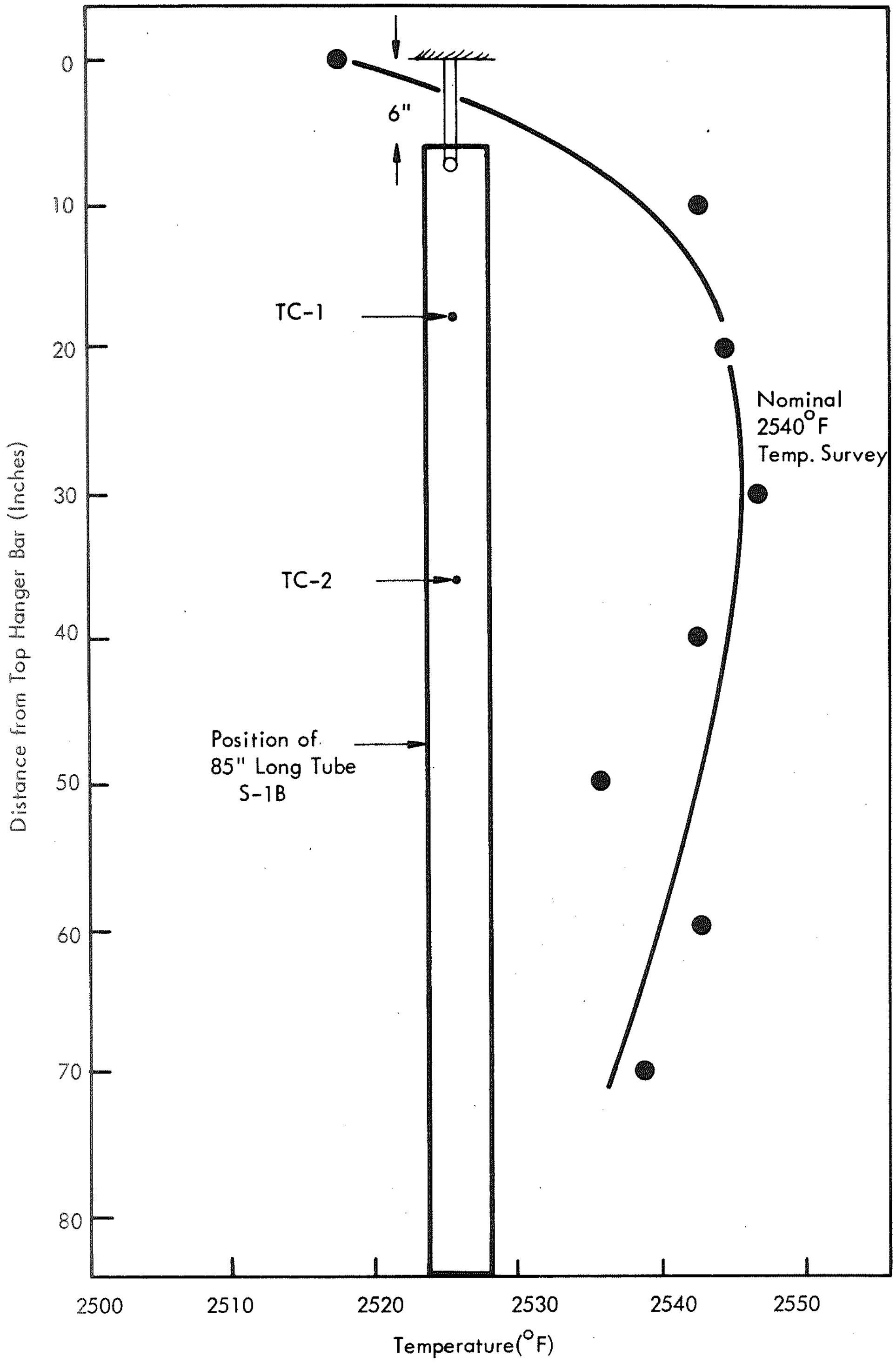


Figure A1-1. Vertical Temperature Distribution of Wah Chang Vacuum Annealing Furnace

4. Industrial Forge
   540-A Cleveland Avenue
   Albany, California 94710

   Hot forging - steam hammer.
   1900 ton drop weight - Erie Foundry Hammer.
   2400°F gas fired furnace.
   Personnel: Owner Operator - D. Hawley

5. Stellite Div. of Cabot Corp., formerly Haynes Stellite Div. of Union Carbide Corp.
   Kokomo, Indiana

   Warm rolling of plate.
   1500 ton separating force Schloemann reversing mill.
   2 high, 31 1/2" dia. rolls, 52" wide.
   1200 horsepower.
   Personnel: Marketing - H. Webb
   Manager of Refractory Products - Culbertson
   Superintendent - Bennett

6. Swepco Tube Corporation
   One Clifton Boulevard
   Clifton, New Jersey

   Press brake tube forming - .425" thick tantalum.
   1500 ton mechanical press - 20' length capacity.
   150 ton closed die sizing press.
   Personnel: Technical Director - J. A. Seme

7. Mech-Tronics Corporation
   1635 N. 25th Avenue
   Melrose Park, Illinois 60160

   Vacuum electron beam welder.
   30 KW, 60 KV, Mechtronics Modified Sciaky Welder.
   Triode gun, power or amperage control.
   15 KW, 60 KV, Sciaky Welder.
   Chamber size (30" deep, 40" long, 50" high)
   Carriage travel 24".
   30" continuous weld made by moving gun and carriage.
   Personnel: Manager - S. DeMuro
   Technical Director - L. McNab

8. The Timken Roller Bearing Co.
Canton, Ohio 44706

Tube reducing using Rotor-roll or cold pilger.
2 roll equipment, Wooster, Ohio factory.
6 1/2" diameter capacity Roto-roll - Aetna Standard.
3" diameter capacity Roto-roll.
Personnel: General Superintendent - R. R. Elsasser
Marketing - J. J. McGram
Plant Manager - A. A. Agnes

APPENDIX 2

Analysis of T-111 Extrusion Defect

APPENDIX 2

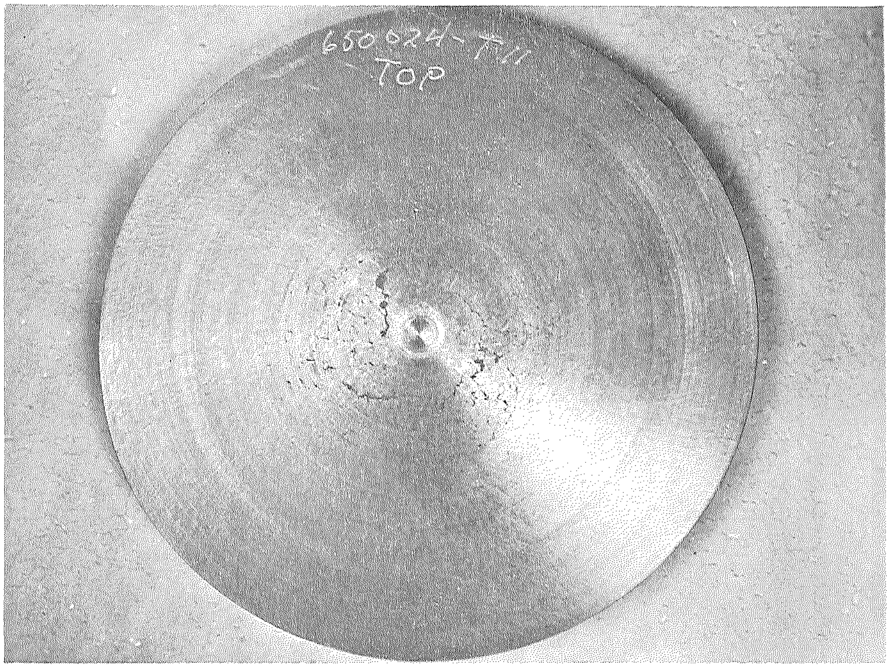
Analysis of T-111 Extrusion Defect

A2.1 Extrusion Reduction Ratio

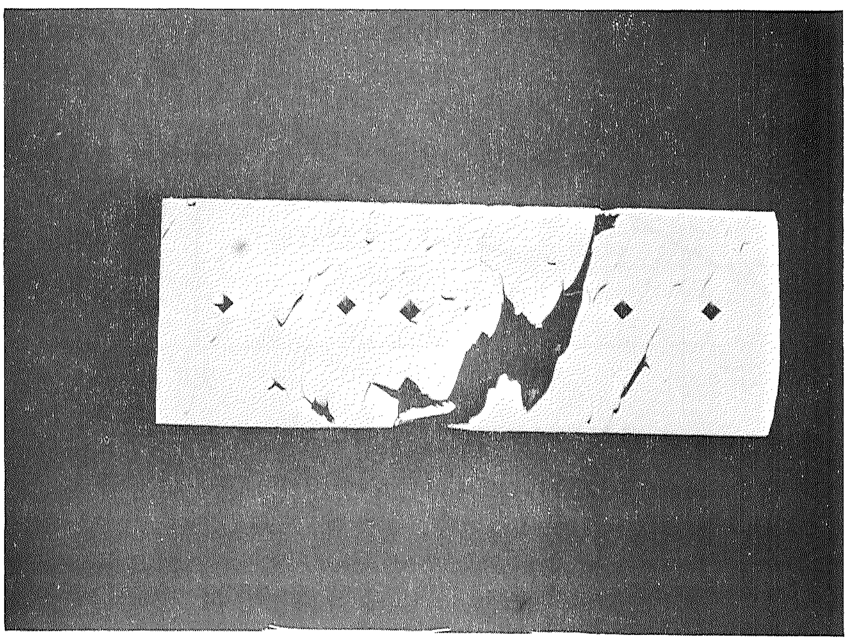
The first solid extrusion of T-111 made for this program developed internal fissures and was not usable. An extrusion cross section and longitudinal slice is shown in Figure A2-1 which indicates a typical center burst fissure. The outside surface of the extrusion was sound as were the nose and tail. The longitudinal distribution of defects showed a gradual increase to a maximum 2/3 the distance from the front. The extrusion, which was required as the initial ingot breakdown step for producing welded tube shell plate, was subsequently remelted and successfully re-extruded. T-111 had previously been extruded several times under identical conditions at Canton Drop Forge by Wah Chang Corporation, so the extrusion failure was unexpected. Of the suspected causes of the failure, (which included ingot porosity, severe alloy inhomogeneity, trace impurities such as nickel, copper or hydrogen, insufficient heating time, and extrusion reduction ratio), the relatively low extrusion reduction ratio of 2:1 was considered the most likely. As discussed by Avitzur*, center burst defects in extrusions are affected by several variables, such as die angle and friction but are strongly dependent on reduction ratio.

Low reduction ratios at a given set of borderline conditions will produce discontinuous flow across the extrusion billet cross section and will result in internal voids commonly called center bursts. Figure A2-2 shows the relationship developed by Avitzur* for central burst tendency as a function of die angle and reduction ratio. A friction factor of 0.5 is assumed from a range of 1.0 to 0 in plotting the curve. Referring to Figure A2-2,the defective extrusion was extruded at a reduction ratio of 2 to 1, through dies with an entry semicone angle of 45°. The re-extrusion of the remelted T-111 was made at a reduction ratio of 3.3:1 with the same die angle

*B. Avitzur, Analysis of Central Bursting Defects in Extrusion and Wire Drawing, Transactions of the ASME, Journ. of Eng. for Industry, Feb. 1968.



6" Diameter Transverse Section



Extrusion
Direction

17,575 Longitudinal Section 5X

Figure A2-1. Sections of Defective 6" Diameter Extrusion

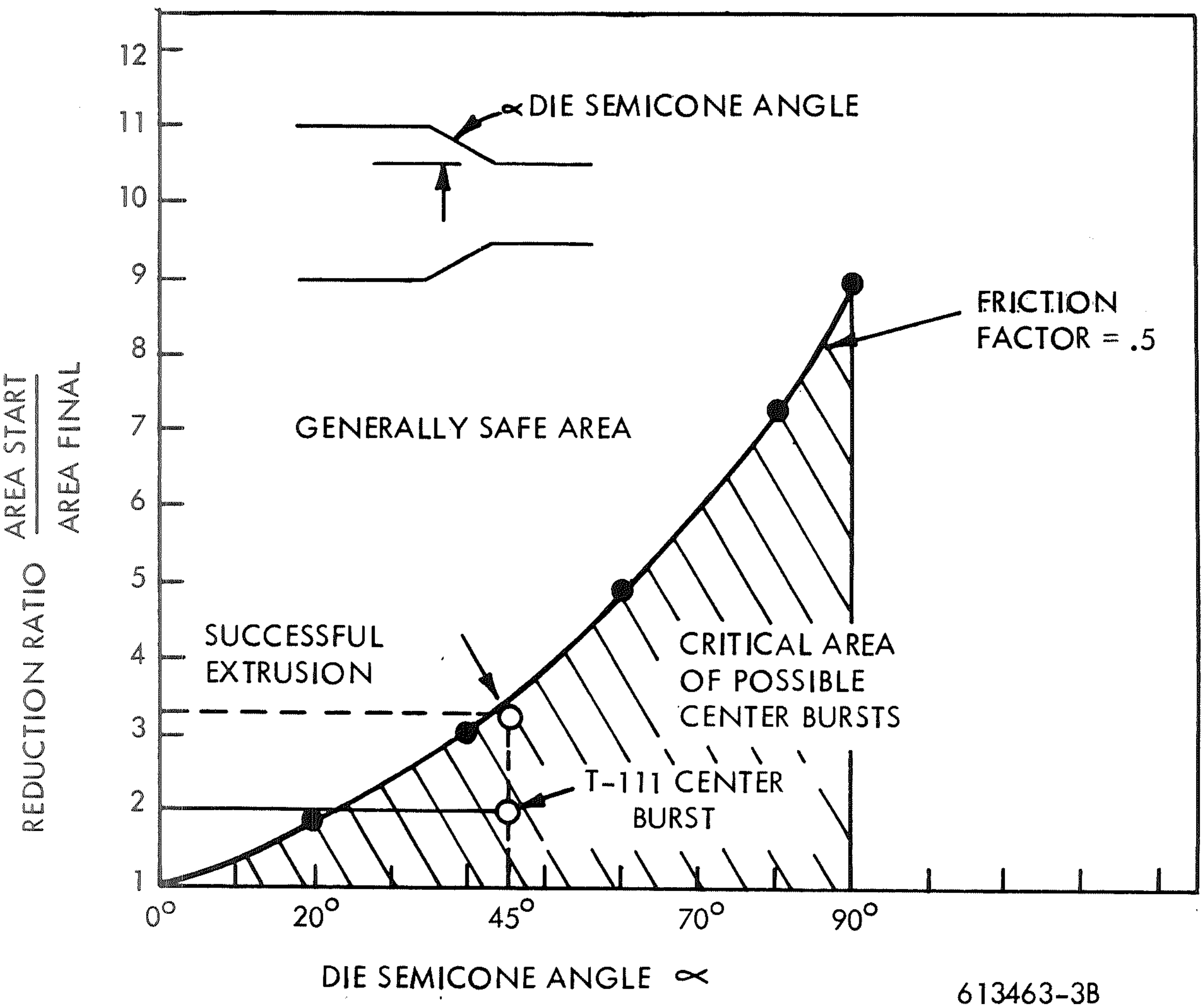


Figure A2-2. Relationship of Die Angle, Area Reduction Ratio, and Central Bursting[1] Tendency and was successful with no internal defects. Center bursting may be due to many other factors including heat to heat variability in mechanical properties as indicated by the several good T-111 extrusions made at a 2:1 reduction ratio prior to the center burst extrusion made for this program. Following the failure, Wah Chang extruded an additional 9 inch billet at a 2:1 reduction ratio with no center burst problem.

Conclusion

In that no distinct differences were noted in starting billet chemistry, morphology, or in the extrusion parameters as compared to previous successful extrusions, we assumed the extrusion ratio of 2:1 was too low to prevent center bursting and a greater extrusion ratio would be desired. For the second extrusion of the remelted T-111, an extrusion ratio of 3.3:1 was used which was as high as possible with consideration to the press capacity and required product size.

Immediately following the initial defective extrusion several ancillary evaluations were made on extrusion related reactions as part of a general corrective action. One evaluation consisted of determining the consequence of a leak in the protective steel can during salt bath heating. Another evaluation was made to determine the effectiveness of tantalum foil and flame sprayed molybdenum protective layers between the steel extrusion container and the T-111 billet. The results are presented in sections A2.2 and A2.3. Because a tantalum foil protective layer was used between the T-111 billet and the steel protective can, there was some doubt that an adequate billet temperature would be obtained in the normal 2 1/2 hour salt bath heating time that was established for the several preceding Wah Chang T-111 extrusions which did not use tantalum foil. To settle the question and provide a guide to the required heating time, thermocouples were imbedded 1 1/2 inches in the base of both the solid and hollow T-111 extrusion billets and the temperature was monitored throughout the salt bath heating. The temperature measurements indicated that the billet heating rate was rapid and 2 1/2 hours was adequate for the 900 lb solid extrusion and a shorter time was sufficient for the hollow billet. The temperature measurements were presented in section 4.2.

A2.2 Corrosion of T-111 in 2300°F Salt Bath

To determine the consequences of a leak in the steel protective container during salt bath heating, 3/8 inch thick specimens of T-111 were exposed in the Canton Drop Forge salt bath at 1, 2, and 4 hours at 2300°F. A carbon steel cylinder was used to hold the specimens and to provide atmospheric protection at the time of removal. The specimen bucket, containing a volume of salt, was removed following the exposure and the salt quickly frozen in place. The solidified salt which is nominally a neutral barium chloride mixture designated as Park Chemical Co. K-90 was then removed by dissolving in hot water.

A hardness traverse of the specimens as shown in Figure A2-3 indicated a distinct hardened zone from .070" to .100" deep on both specimens. Metallographic sections, Figure A2-4, indicated a microstructural change at the approximate distance of the rapid increase in hardness. Significant structure changes were observed at the specimen centerline, Figure A2-5, indicating contamination beyond the obvious hard case. Although the contaminated layer was not chemically analyzed, the rapid diffusion of the contamination is indicative of interstitial contamination such as oxygen or nitrogen. A .107" thick bend test specimen was made of a 2 hour exposure specimen by grinding .135" from both exposed surfaces to remove the bulk of the contamination. A 1T bend test at -320°F, which normally produces a fully ductile 90° bend, resulted in a brittle fracture with no measurable plastic deformation indicating severe embrittlement through at least .135" of surface.

The salt bath exposure indicated that a leak tight protective container is definitely required to prevent excessive material losses and possible extrusion failure.

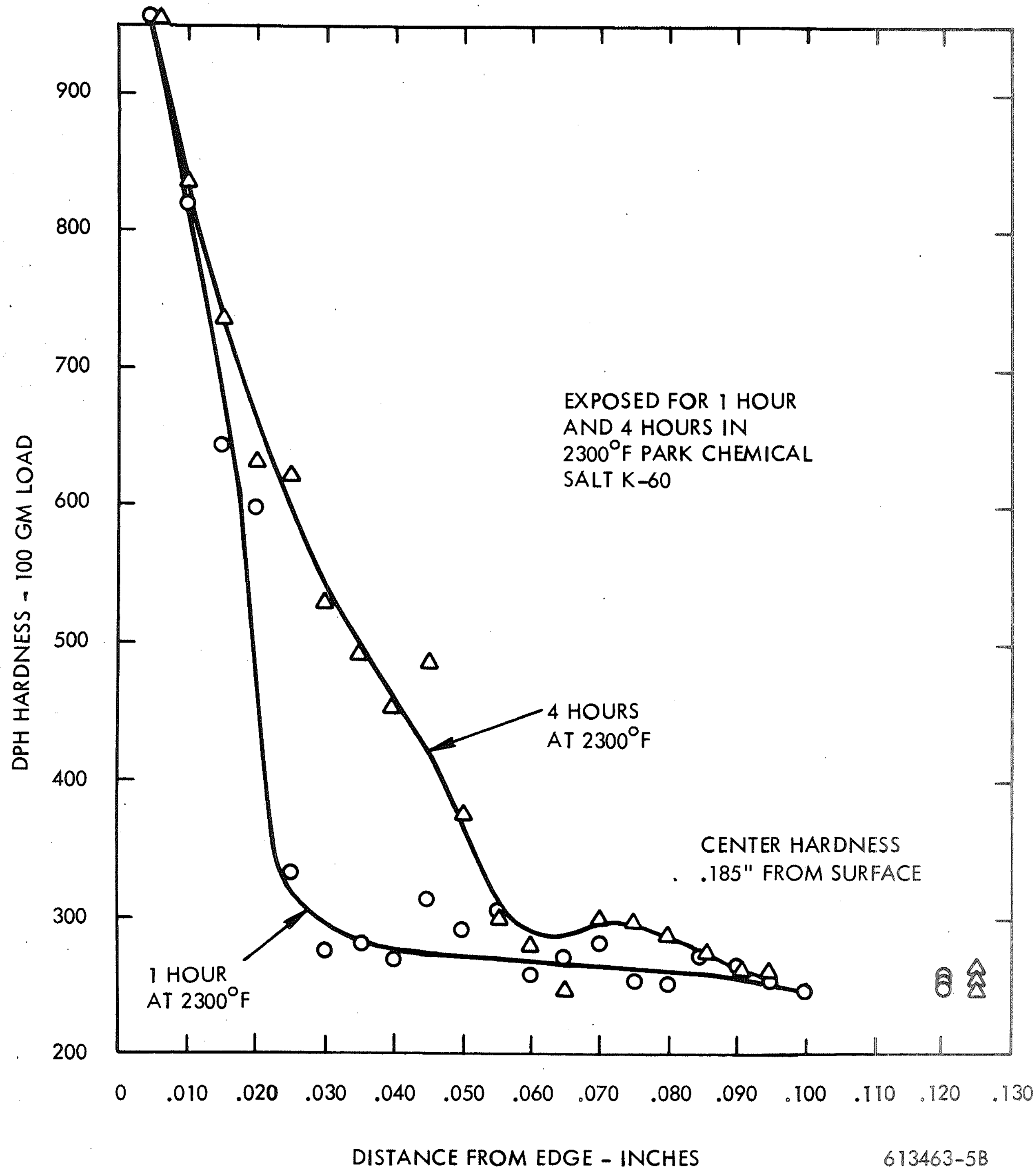
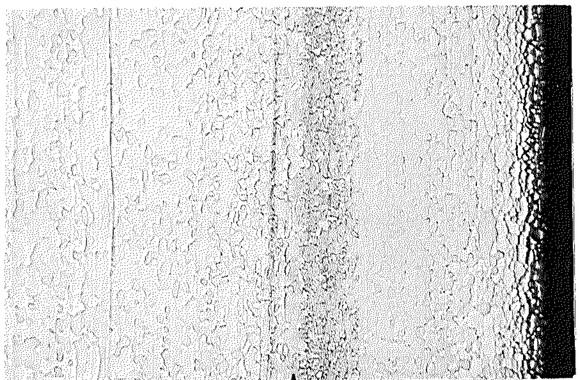


Figure A2-3. Hardness Traverse from Surface to Center of 3/8" Thick T-111 Alloy Specimens After Exposure to Park Chemical Salt K-60 at a Temperature of 2300°F

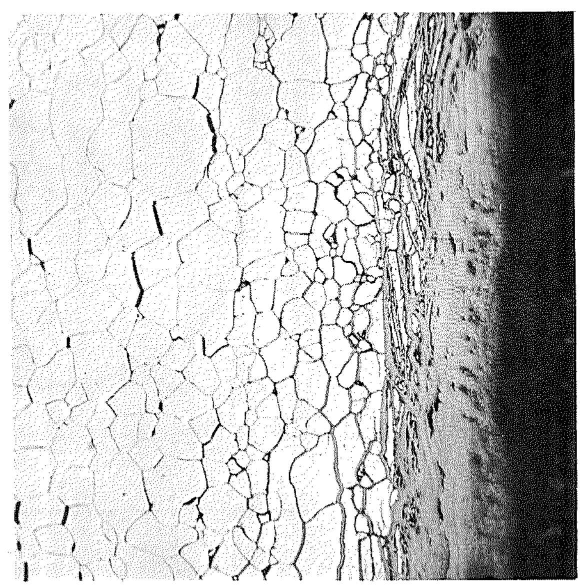
1 Hr 2300°F



50X

4 Hrs 2300°F

Enlarged Area of Surface

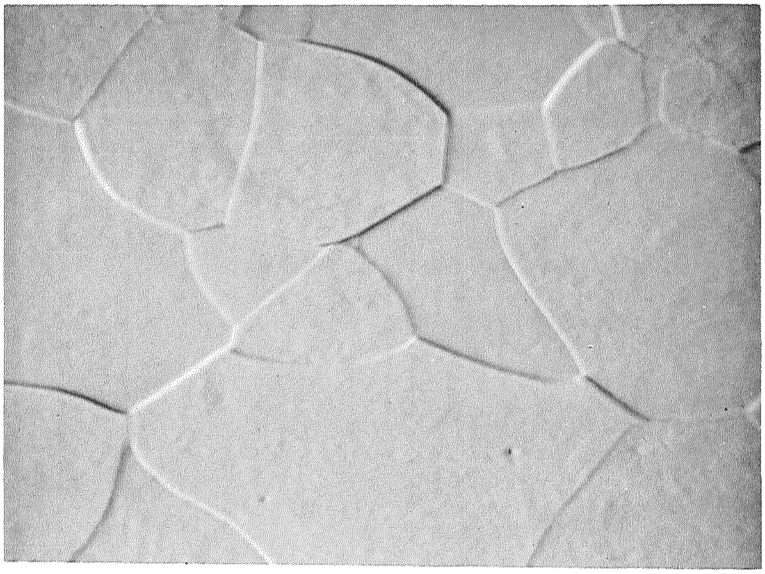


Transverse sections of .375" T-111 Test Coupons exposed to BaCl salt bath at 2300°F for times indicated.

Salt bath at Canton Drop Forge Corp., Canton, Ohio.

Park Chemical K-60 Neutral Salt

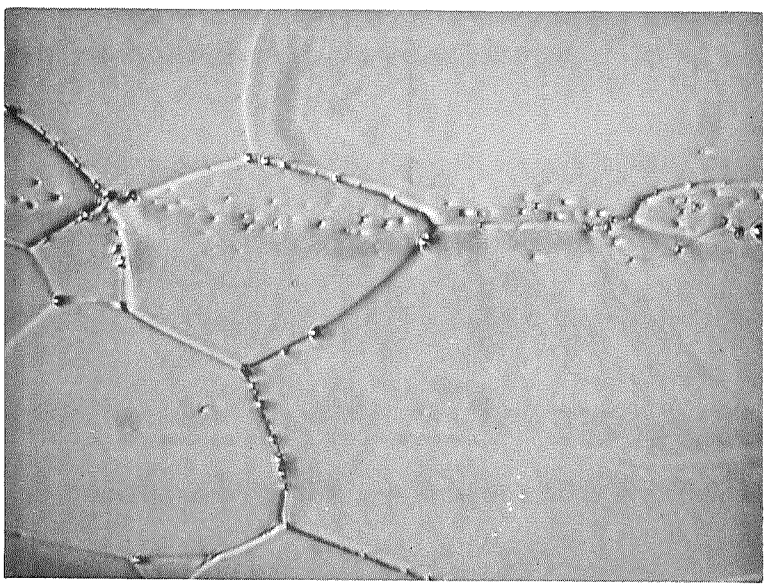
Figure A2-4. Sections of T-111 Contaminated by 2300°F Salt Bath



1 Hr, 2300°F

Specimen Centerline
.185" from Surface

15,550 1500X



4 Hrs, 2300°F

Specimen Centerline
.185" from Surface

15,530 1500X

Figure A2-5. Center Sections of T-111 Contaminated by 2300°F Salt Bath

A2.3 Tantalum Foil Contamination Barrier

A screening experiment was performed to determine the relative effectiveness of tantalum foil and flame sprayed molybdenum in preventing contamination of the T-111 from the 1020 steel container. Interstitial migration, particularly carbon from the 1020 steel, is expected at 2300°F. Four conditions were evaluated following a 5 hour exposure at 2300°F.

(a) 1 layer of 0.0025" tantalum foil
(b) 2 layers of 0.0025" tantalum foil
(c) flame sprayed molybdenum - 0.002"
(d) no barrier - bare T-111

Figure A2-6 shows the details of the diffusion couple which although designed "inside out" as compared to an extrusion billet, maintains the same T-111 to steel volume ratio as a 9 inch diameter T-111 billet clad with 1/4 inches of carbon steel. By placing the carbon steel on the interior, the greater thermal expansion of steel maintained contact between the layer components. The capsule was welded in a helium welding chamber and the vacuum sealing plug was closed by electron beam welding at 5×10^{-5} torr, simulating the conditions in a vacuum sealed extrusion container.

Metallographic sections of the steel to T-111 interface for the four test conditions are shown in Figure A2-7. In the low magnification section, the most pronounced difference between the sections is the lack of pearlite formation in the flame sprayed molybdenum and in the bare T-111 sections. Apparently, the single and double layers of tantalum foil effectively prevent the migration of carbon from the 1020 steel to the T-111. A microhardness traverse of the T-111 section at the steel to T-111 interface, Figure A2-8, indicates a higher hardness for the flame sprayed molybdenum specimen and the bare T-111 specimen to a depth in the T-111 of approximately .010 inches. In referring to Figure A2-7, an interesting observation is that carbon transfer from the 1020 steel to T-111 occurred across the large gap shown in the bare T-111 section. Transfer by CO, CO_2, or CH_4 formation and subsequent decomposition at the T-111 surface is possible in the sealed system.

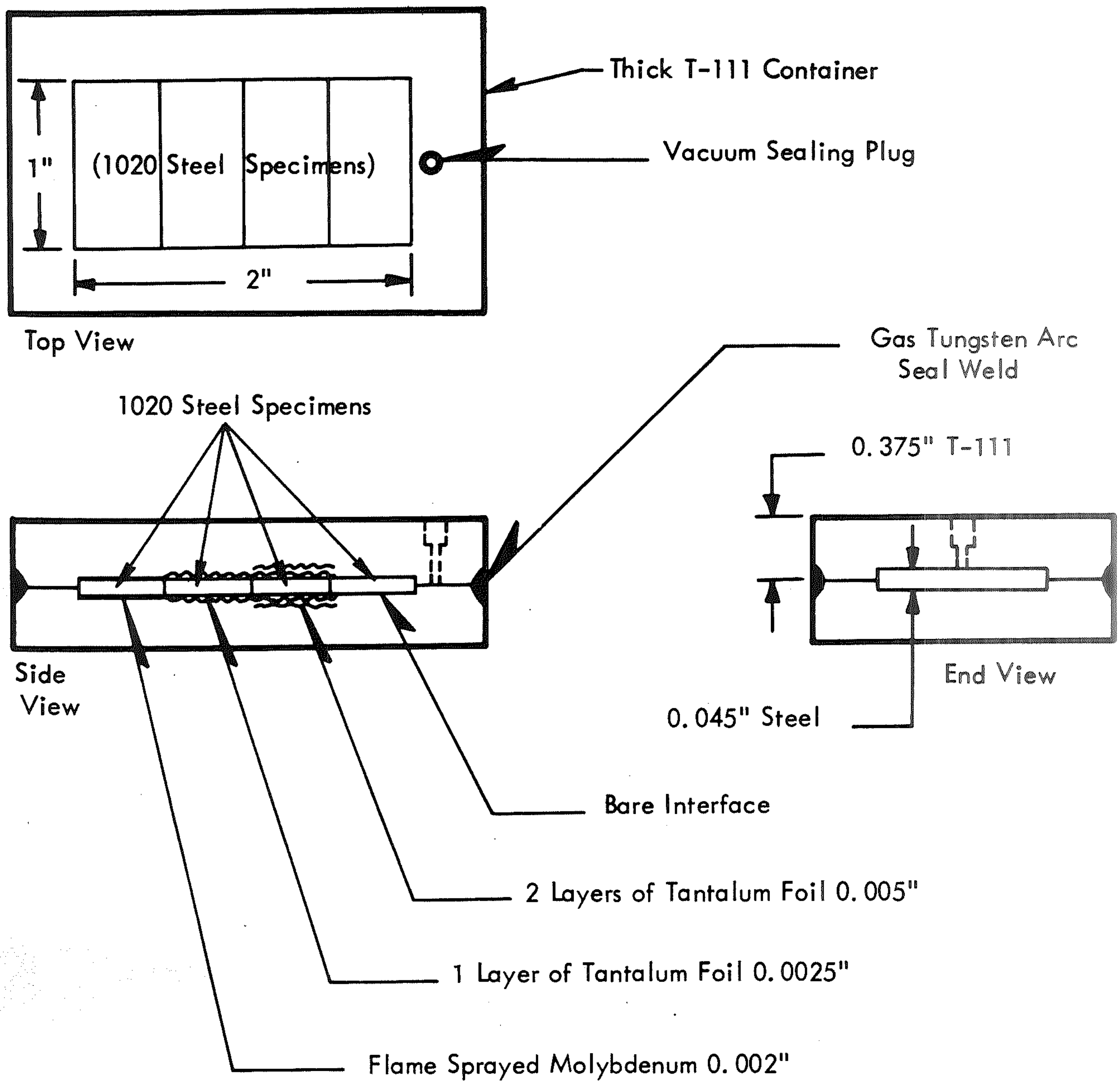
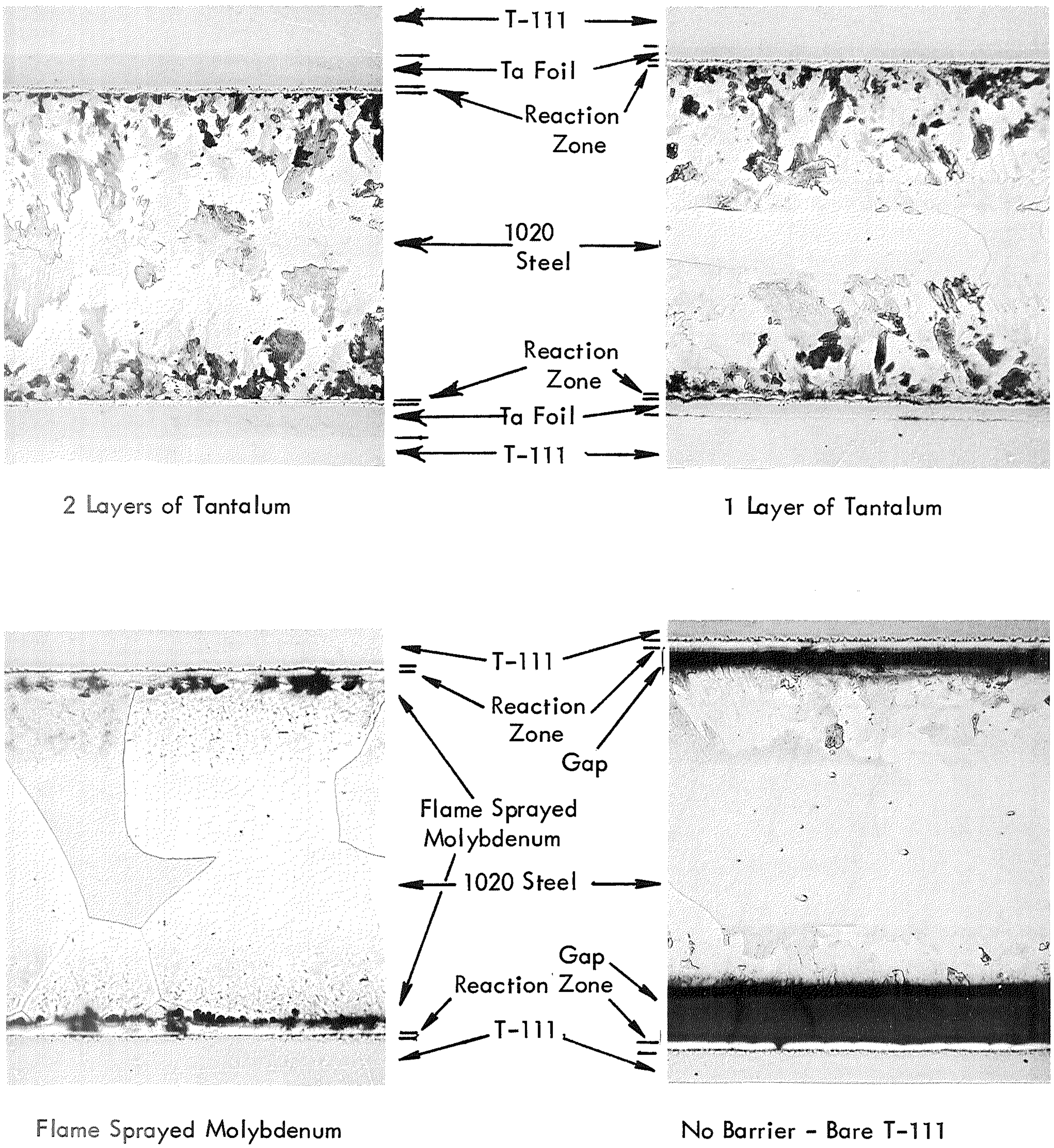


Figure A2-6. Carbon Steel - T-111 Diffusion Couple for Evaluation of Barrier Layer Performance



18,378 50X

Figure A2-7. Microstructural Changes in Mild Steel - T-111 Alloy Couples as a Function of Various Barrier Layers Following 5 Hours at 2300°F

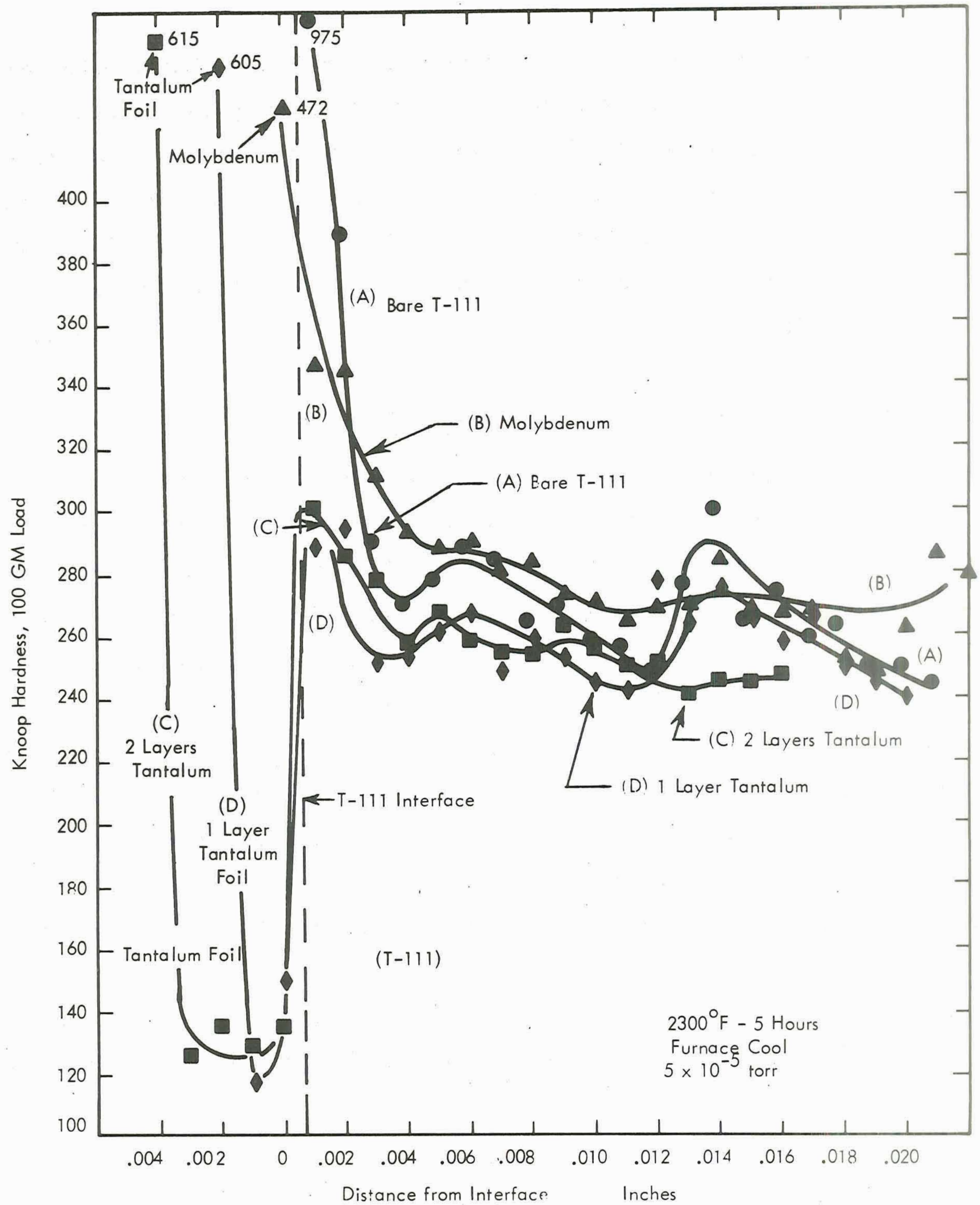


Figure A2-8. Hardness Traverse of Mild Steel to T-111 Interface in a Diffusion Couple as a Function of Various Barrier Layers

Based on these observations, tantalum foil layers were judged to be effective in minimizing interstitial contamination of T-111 from a steel container. Flame sprayed molybdenum was judged to be particularly ineffective. The relative value of any of the diffusion barriers must be weighed in view of the normal 0.050 inch to 0.100 inch surrace removal following extrusion since the major contamination is confined to within 0.005 inches of the surface. One layer of tantalum foil was included in the assembly of the extrusion billet which was a compromise between contamination protection and heat transfer during heating to extrusion temperature.

A2.4 Selection of Extrusion Billet Protective Container Material

Mild steel (1020 designation) was used as the billet protective container. Experience has indicated that this material is satisfactory for salt bath heating, where scaling is suppressed, up to 2300°F. At this temperature, a shallow intermetallic reaction zone is produced on the T-111 extrusion which must be removed during conditioning. Killed steels have excellent welding properties and also have less tendency for interstitial migration from and through the steel protective layer. Stainless steel or nickel base alloy extrusion containers, which would provide increased strength and scaling resistance at the 2300°F extrusion temperature, were not considered because of possible grain boundary embrittlement of the T-111. Nickel embrittlement has been observed at temperatures over 2000°F. Nickel-hafnium eutectic reactions at hafnium rich grain boundaries are postulated as the failure mechanism. All protective extrusion containers used in this program were fabricated from low carbon 1020 steel.

Molybdenum extrusion billet containers, either brazed or welded,would provide good performance at 2300°F at increased cost and permit increasing the extrusion temperatures to 3000°F.

APPENDIX 3

Thick Section T-111 Multi-Pass Inert Gas Tungsten Arc Welding

APPENDIX 3

Welding Investigation of Thick Section T-111 Plate

The initial plans for producing the longitudinally welded tube shells included evaluating both electron beam fusion welding and gas tungsten arc welding using T-111 filler metal. Combination welds such as electron beam root passes and GTA filler metal additions were also considered, but not actively explored. The relative merits of electron beam and gas tungsten arc welding are listed as follows:

Electron Beam

Advantages	Disadvantages
1. Minimum distortion	1. Precision weld tooling required
2. Low total heat input	2. Precision part fit up required
3. Small total weld size	3. Inspection is difficult
4. Single pass capability	4. Possible loss of high vapor pressure alloy constituents
	5. Difficult weld repair

Gas Tungsten Arc with Filler Wire

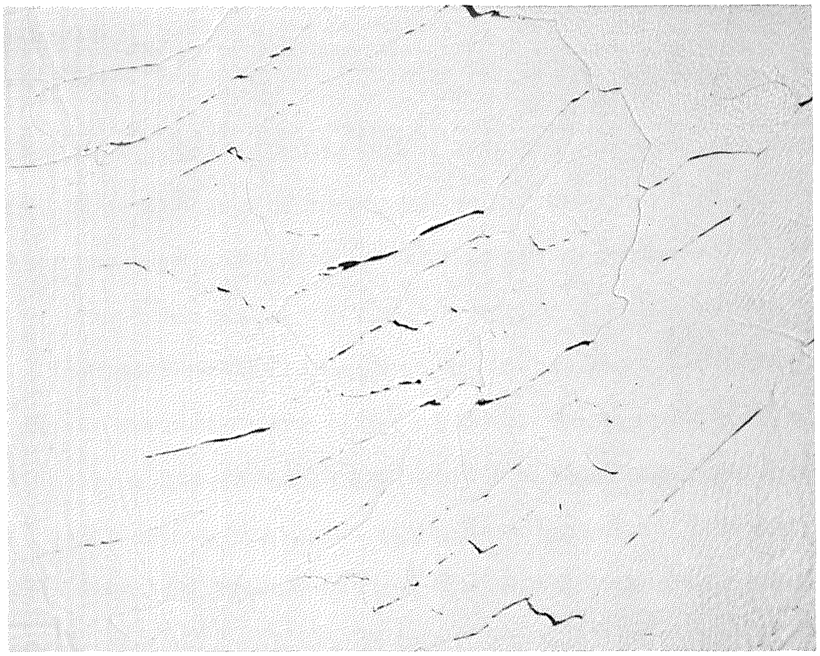
Advantages	Disadvantages
1. Minimum tooling required	1. High total heat input
2. Part fit up not critical	2. Severe distortion
3. Weld repair is possible	3. Large total weld size
4. Good choice for "one of kind" process	

Multipass manual or semi-automatic gas tungsten arc welding was originally favored because the advantages appeared to outweigh the disadvantages for this application. Also, more experience in welding T-111 had been accumulated by the gas tungsten arc process as typified by the previous successful GTA welding of T-111 sheet (.035 inch thick) and plate (.375 inch thick) (NAS 3-2540). Customary microscopic examination and full weld section bend tests of both the sheet and plate had not indicated any welding difficulties.

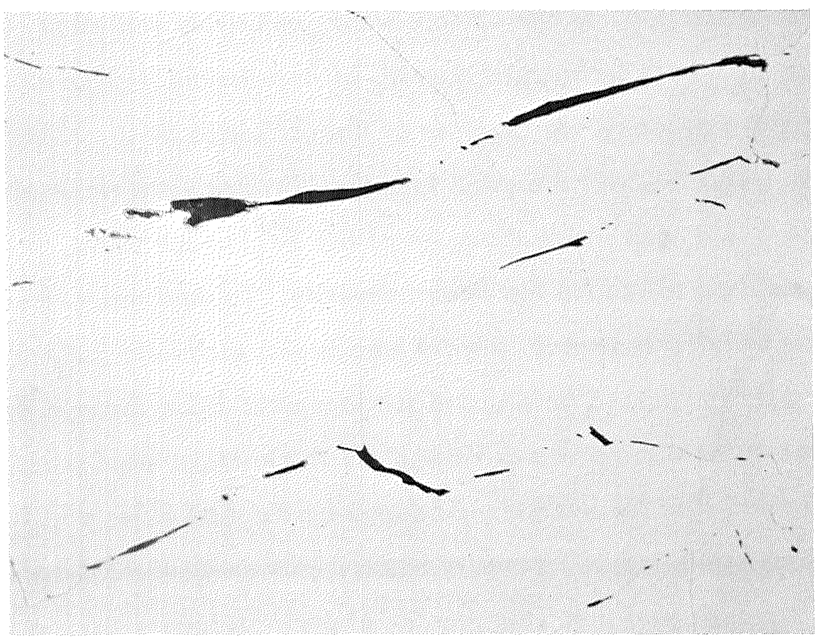
The tube shell welding for this program, however, required single surface welding of thicker section T-111 plate, (.425 inch thick), as compared to the less severe alternating weld passes used in the previous investigation. Examination of the first GTA welds made from one side of .425 inch thick plate indicated grain boundary fissures through the entire weld section except the surface weld pass. Figure A3-1 shows a typical transverse GTA weld section with grain boundary fissures. This problem of weld fissures in T-111 was evaluated in some detail in terms of ingot composition, weld geometry and welding parameters and is presently considered as a tendency in all gettered high strength refractory metal alloys and is not peculiar to T-111. Filler metal plate welds were made in T-222 (Ta-9.6w/o W-2.4w/oHf), ASTAR-811C (Ta-8w/o W-1w/o Re-.7w/o Hf-.025w/o C), and FS-85 (Cb-27w/o Ta-10w/o W-1w/o Zr), and similar underbead, grain boundary fissures were observed. Plate multipass filler welds made in Ta-10W, an ungettered alloy, were not fissured. The sensitivity is related to joint thickness as single pass thin section welds have not displayed this characteristic. As shown in Figure A3-2, which outlines the various weld geometries and alloy heats evaluated, alternating passes from both surfaces of a weld, as in a double J groove, greatly decreases the fissure size and frequency. In the case of the .375 inch thick double J welds made in a previous program, (NAS 3-2540) the grain boundary fissures were reduced to the limits of detectability. The 4 3/4 inch inside diameter of the T-111 tube shells prevented the use of double side welding to reduce the fissure severity. To determine when the fissures occurred in a multipass welding sequence, a weld groove was gradually built up from a root pass to complete 11 passes in stepped fasion, and sections were made through the gradually increasing weld thickness to determine if a critical plate thickness or weld pass size existed. The evaluation indicated that fissures were produced by the first filler pass in the root pass weld metal. The number and size of fissures gradually increased with the number of weld passes. Additional work has indicated that* the number of grain boundary fissures is reduced by a larger number of lower heat input filler passes. In the weld evaluation done for this program approximately 9 to 11 passes were used for a single "V" plate weld in

---

*R. E. Gold and G. G. Lessmann, Influence of Restraint and Thermal Exposure on Welds in T-111 and ASTAR-811C, Final Report WANL-PR-(VVV)-001, NASA CR-72858, March, 1971



18,754 50X



18,754 200X

Wah Chang Plate & Wire

50° "V"
Annealed 1 Hr. at
3000°F Prior to
Sectioning

Figure A3-1. Transverse Sections of GTA Filler Weld in T-111 Plate

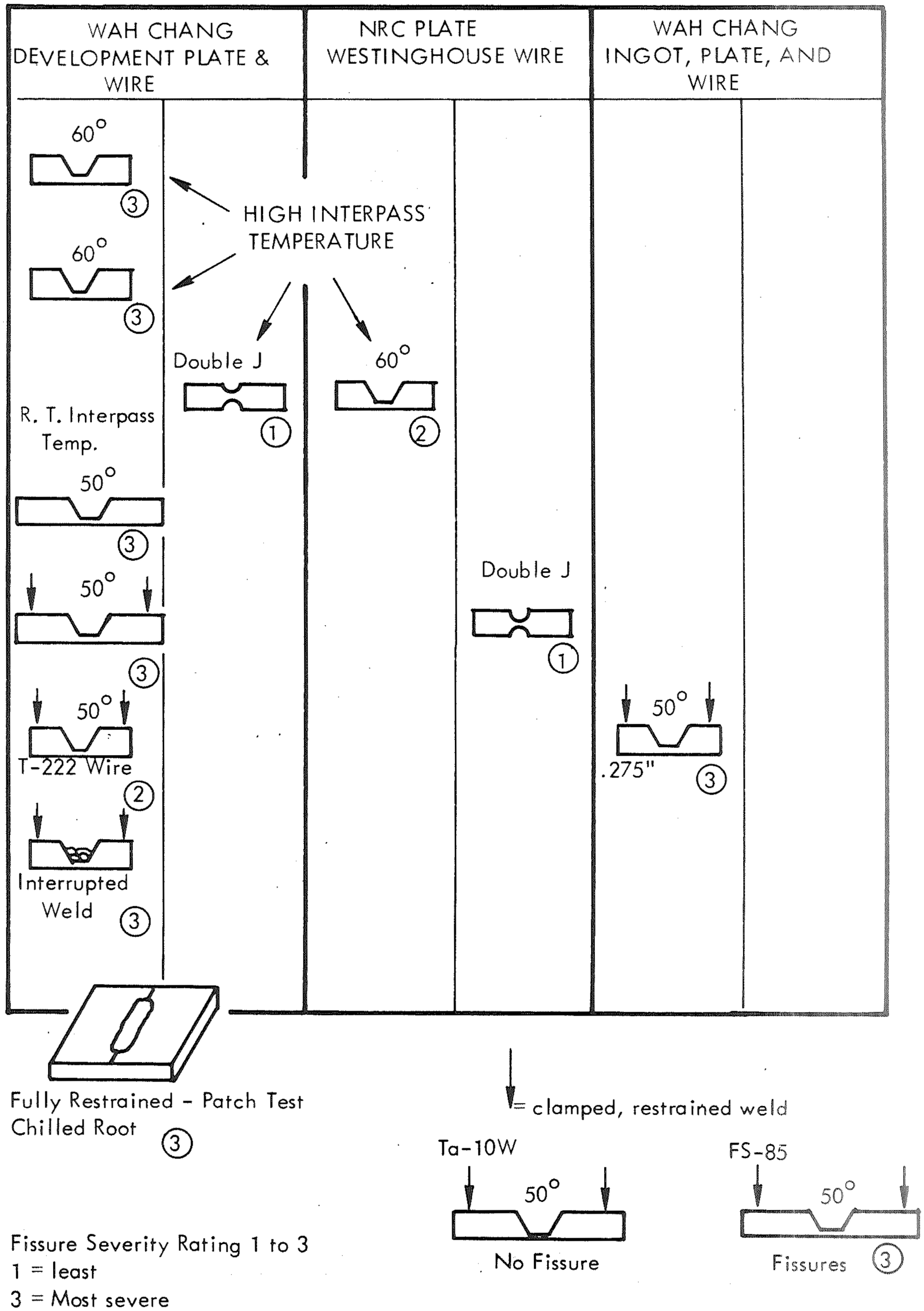


Figure A3-2. Variations in T-111 Manual GTA Plate Welding Evaluated

.425 inch thick T-111. All GTA plate welding was done in a high purity, vacuum purged, helium atmosphere welding chamber.** Water vapor and oxygen in the chamber atmosphere were continually monitored and maintained at levels below 5 ppm O_2 and 10 ppm during welding. All weld specimens were cleaned and pickled prior to welding and only refractory metal alloys were used in contact with the weld specimens. Chemical analyses were made of the T-111 weld specimens with particular emphasis on spectrographic analyses for trace impurities such as copper and nickel and no discrepancies were observed in ingot analysis or in trace impurities which could be related to the welding difficulties.

At present, there is no conclusive mechanistic analysis of the underbead grain boundary fissures in T-111 plate welds. The fissures never occur in the surface weld bead, but rather in the preceding weld passes or in the base metal if a sufficiently large grain size exists. Temperature measurements in the fissured area are in the range of 2800°F during welding. The general analysis is that the thermal strain produced by the last welding pass cannot be accommodated by the grain boundaries at high temperature and fissuring occurs.

Since a practical solution to underbead cracking in GTA filler welds was not obtained, electron beam welding was selected as the reference tube shell welding process early in the development program. The single pass electron beam welding process selected provided no opportunity for thermal straining large grained, previously deposited weld metal. In addition, the small overall size of the electron beam weld reduced the area and possibly the magnitude of the thermal strain. Throughout the welding program, no sign of weld fissures were observed in electron beam welds.

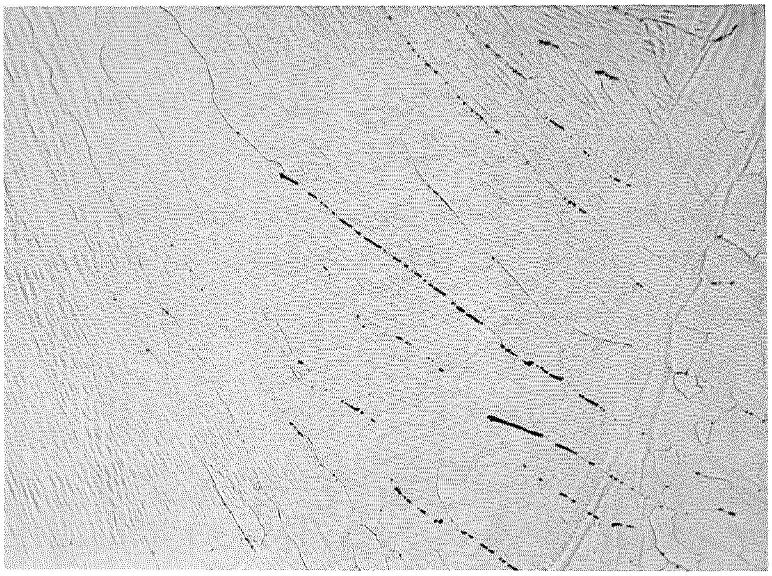
**D. R. Stoner and G. G. Lessmann, <u>Measurement and Control of Weld Chamber Atmospheres,</u> Welding Journal August 1965.

Tube Reduction Behavior of Gas Tungsten Arc Welded Small Tube Shell

Electron beam welding, normally being a rigidly fixtured, chamber welding process, precludes application to a large variety of weld fabrication processes including manual filler wire techniques. To determine the feasibility of using the multipass gas tungsten arc welded process on thick T-111 sections that undergo subsequent cold forming operations, a 5 inch length of tube shell, which was groove machined to remove an imbedded thermocouple, was repaired using multipass GTA filler wire welding. A total of 13 weld passes were used of gradually increasing power from 255 amps and 21 volts to 340 amps and 26 volts. The manual welding was done in a vacuum purged, impurity monitored, helium atmosphere welding chamber. The GTA welded tube shell was then tube reduced in 3 passes from 5 3/4 inch OD by .580 inch wall to 3 inch OD to .080 inch wall and vacuum annealed 1 hour at 3000°F between passes. The purpose of the tube reduction of the GTA welded section was to determine the effect of the reduction process on the GTA weld fissures.

Figure A3-3 compares a section of the as-welded tube shell weld metal to the 95% reduced sections. The weld fissures are apparently collapsed and bonded by the combination of tube reduction and vacuum annealing. There was no evidence of fissure enlarging during the working process.

As well as demonstrating the feasibility of tube reducing GTA welded sections, the evaluation demonstrated the extremely high room temperature ductility of T-111 since the built-in weld defects did not propagate during the cold working process.



20,578A

50X

Fusion Line

As Welded Tube Shell

5.750" OD x .570" wall

21,171 Fusion Line

100X

Tube Reduced 92%, 65%, 52%

3000°F - 1 hr. Interpass Anneal

Figure A3-3. Transverse Sections of Manual GTA Filler Weld in T-111 Tube Shell

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